VOL. LV, NO. 5, MAY, 1960

THE Journal OF THE AMERICAN LEATHER CHEMISTS ASSOCIATION

PUBLISHED MONTHLY BY
THE AMERICAN LEATHER CHEMISTS ASSOCIATION
PUBLICATION OFFICE, EASTON, PA.

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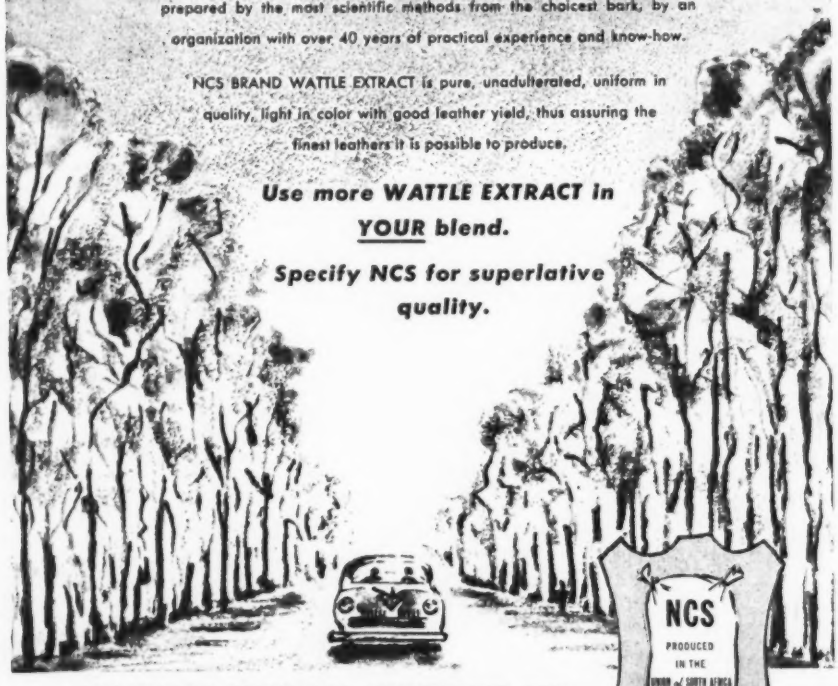

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The Journal

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Vol. LV MAY 1960 No. 5

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SUBSCRIPTION - - MEMBERS $12.00 A YEAR - - NONMEMBERS $15.00

Published Monthly by

The American Leather Chemists Association

Entered as Second-Class Matter at the Post Office, Easton, Pa.
Acceptance for mailing at special rate of postage as provided for in section 1103, Act of October 3, 1917, authorized July 16, 1918

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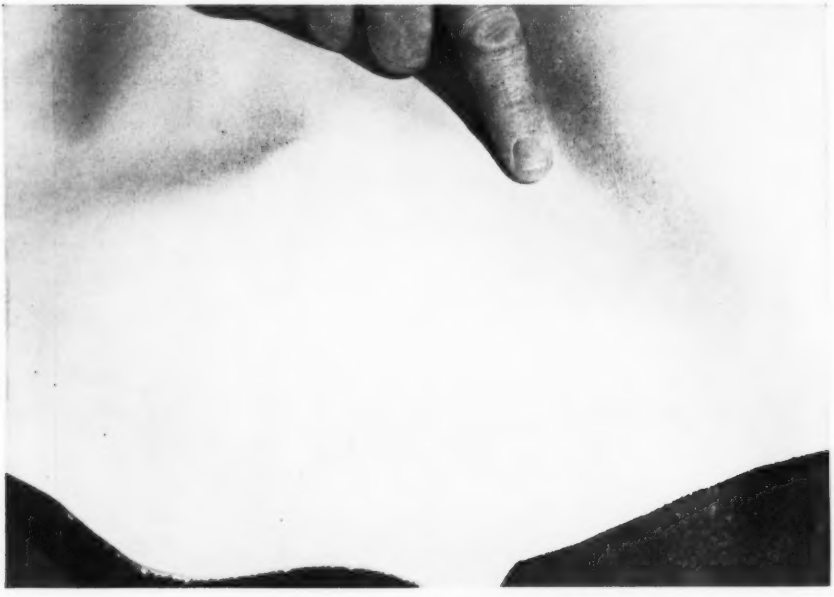

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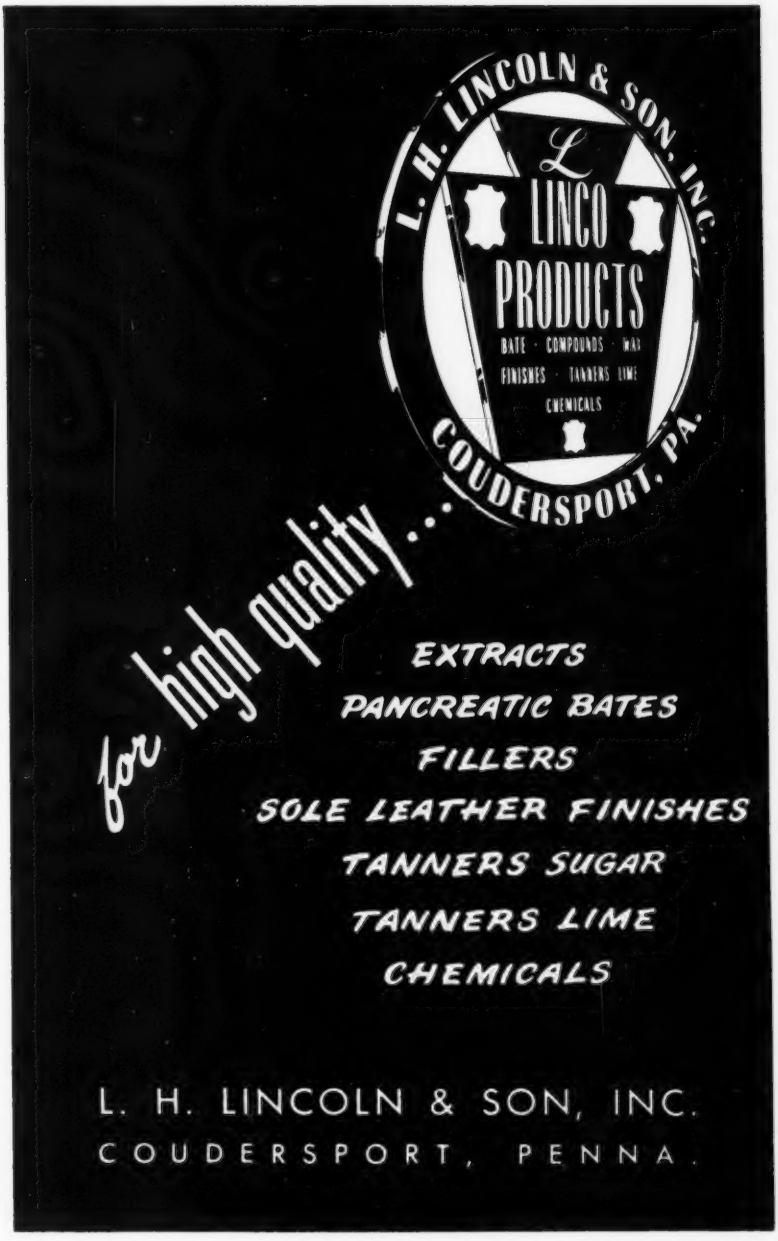
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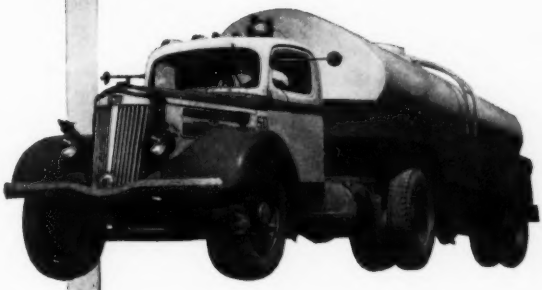

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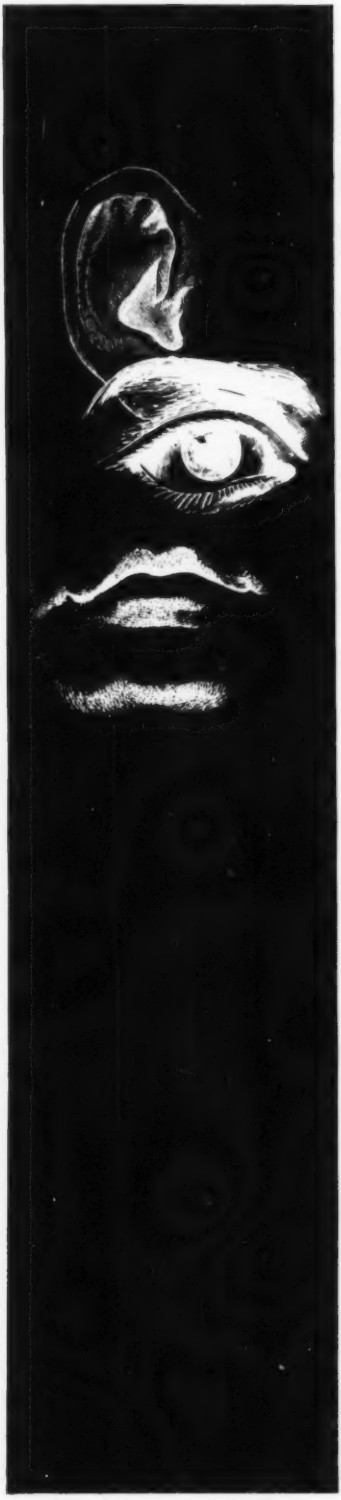

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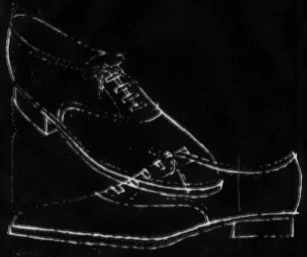

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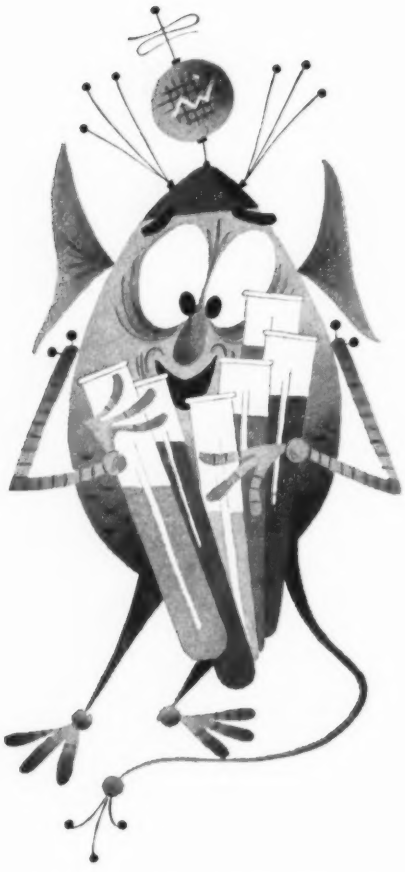

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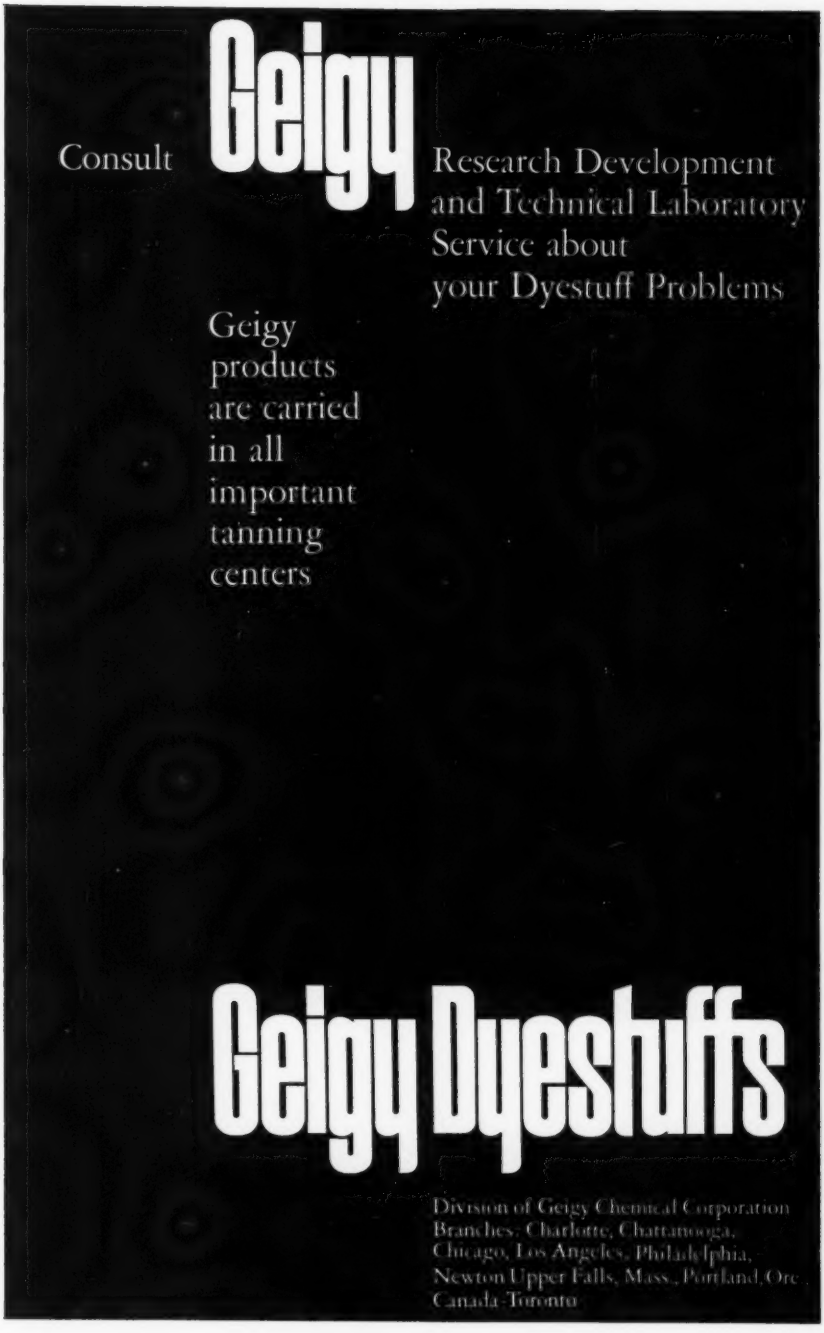
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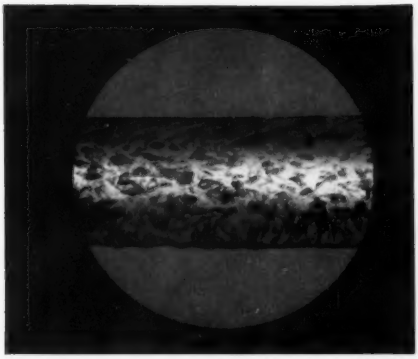

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PAPER ELECTROPHORESIS AND CHEMICAL ANALYSIS OF EXTRACTS OF STEERHIDE CORIUM AND OF WHOLE STEERHIDE

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ABSTRACT

The present study was undertaken to increase our knowledge of those proteins of hides which are soluble in aqueous salt solutions. The amounts of tota protein and of hydroxyproline-containing material present in extracts obtained by successive extractions of bovine corium with 5% NaCl, 0.1*M* Na_2HPO_4, and 0.1*M* citrate buffer (pH 3.6) were determined; paper electrophoretic examinations of the extracts for bromphenol blue-staining proteins (serum proteins) and for glycoproteins were also carried out. Extracts obtained from whole steerhide by extractions with 5% NaCl, 0.1*M* citrate buffer (pH 3.6), 0.1*M* Na_2HPO_4, half-saturated $Ca(OH)_2$, and 10% $CaCl_2$ were similarly analyzed and examined electrophoretically. Citrate buffer was outstanding in the extraction of hydroxyproline-containing protein (soluble collagen), while half-saturated $Ca(OH)_2$ was the best extraction solution for total proteins and for glycoproteins.

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INTRODUCTION

It has long been known that proteins soluble in aqueous salt solutions are present in the skin. Such soluble proteins are of interest to the practical tanner, since their degree of removal from the hide may affect the quality of the leather produced. In enzymatic unhairing, removal of such proteins is probably also of great importance.

Since large quantities of aqueous salt extracts from successive extractions of steerhide corium with three solvents were available from a previous preparation of fibrous collagen (1), it was decided to do some preliminary analyses of these solutions and to investigate them electrophoretically. In order to investigate a larger number of extracting solvents it was later decided to run

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small-scale extractions of whole steerhide with these three salt solutions and also with $CaCl_2$ and $Ca(OH)_2$ solutions. The results of these preliminary experiments are given in this paper.

A number of other investigators have previously studied the salt-soluble proteins of hides. Their work has been recently extensively reviewed (2,3,4). Therefore, only those papers especially pertinent to the present work will be discussed here.

EXPERIMENTAL METHODS

Preparation of extracts of steerhide corium.—These extracts were obtained in connection with the preparation of a purified collagen from steerhide corium; experimental details are described in a previous paper (1). The extracts were kept at −20° C. until analyzed. "Dialyzed extracts" were prepared by dialysis in open cellulose dialyzer tubing overnight against water. For the paper electrophoretic examination it was necessary to concentrate the solutions, since the staining techniques used in paper electrophoresis are not sensitive at the very low concentrations of protein present in the extracts. Concentration was carried out in a Rinco Rotating Evaporator under 10-mm. vacuum with the evaporation flask heated in a water bath at 25°–30° C. (bath temperature). The increase in salt concentration and the low solubility of some of the proteins made it impossible to increase the protein concentration to the desired level. The solutions were therefore concentrated slightly greater than tenfold, the precipitated salt and protein were filtered off, and the filtrate was made up to a volume one-tenth that of the original unconcentrated extract.

Preparation of extracts of whole steerhide.—A steerhide from a freshly slaughtered steer was fleshed, frozen for a week, and then cut into ½" squares. The following solutions were used as extracting solvents: 5% NaCl, 0.1*M* citrate buffer (pH 3.6), 0.1*M* Na_2HPO_4, half-saturated $Ca(OH)_2$, and 10% $CaCl_2$.

One hundred-gram portions of the hide were soaked in 300 ml. of each of the solutions at 7° C. for 16 hours. The solutions were not stirred during the extraction period. When the extraction was completed, the solutions were filtered through Whatman No. 1 paper. Each 100-g. portion of hide pieces was again covered with 300 ml. of the same extracting solvent and kept at 7° C. for four hours. The resulting extracts were filtered. A third extraction (16 hours) was similarly carried out. After filtration, each extract was kept at −20° C. until it could be concentrated, analyzed, and examined by paper electrophoresis.

The steerhide extracts were concentrated by dialysis against Carbowax 20M (a polyethylene glycol preparation) (5). During the dialysis most of the salt and nonprotein-nitrogen compounds, along with other diffusible

solids present in the extracts, were removed. The solutions showed increased cloudiness on concentration, but there was no precipitation of protein. In actual practice the dialysis of a 100-ml. aliquot of the original extract was carried out at 7° C. until only 2 to 4 ml. remained; this solution was then made up to a volume of 10 ml. and was stored in the freezer at −20° C. The concentrated solutions represent a tenfold concentration of the original solution, but only approximately 2 to 4% of the amount of nonprotein nitrogen and of the salt present in the original solutions is present in the concentrated solutions. While the freshly prepared concentrated solutions did not show any precipitate, after storage at −20° C. a slight precipitate was present in some of the extracts. For analysis an aliquot containing both solution and precipitate was used.

The extracted hide samples were examined microscopically after fixing in formaldehyde, sectioning, and staining with haematoxylin and eosin.

Methods of chemical analysis.

Total nitrogen.—The corium extracts, both before and after dialysis, and the original corium split were analyzed for total nitrogen by the usual macro-Kjeldahl procedure. For the concentrated corium and concentrated whole hide extracts, a micro-Kjeldahl procedure, which involved determination of the optical density of the solution after Nesslerization, was used (6).

Hydroxyproline.—A modified Neuman-Logan method of analysis for hydroxyproline (7) was used for the analysis of the extracts and of the corium split. The dialyzed corium extracts were hydrolyzed 8 hours in 6*N* HCl, concentrated *in vacuo* to a small volume, and neutralized before analysis.

Paper electrophoretic examination.

Determination of serum proteins.—Paper electrophoresis was carried out on the concentrated extracts with a Spinco Model R Paper Electrophoresis Apparatus and an Analytrol, with the use of the alcoholic bromphenol blue–staining method (Spinco Procedure B) recommended by the manufacturer for human serum (8).

The bands of the concentrated extracts were too light to be quantitatively evaluated on the Analytrol, although they were quite evident to the eye; the papers were therefore eluted with 0.5% Na_2CO_3, and the optical density of the eluates read at 590 mμ on the Beckman Spectrophotometer as recommended by the manufacturer for small amounts of proteins.

For a standard of comparison normal bovine serum (Nutritional Biochemicals Co.) was used. It gave an electrophoretic pattern of four bands (one albumin and three globulins). The albumin band was eluted with 0.5% Na_2CO_3, and the optical density at 590 mμ of the serially diluted eluate was

determined. A standard curve relating optical density to relative albumin concentration was thus obtained for the albumin–bromphenol blue complex.

The absolute amount of albumin present in the bovine serum was determined by analysis for albumin according to the procedure recommended by Varley (9). It should be pointed out that any method of determination of serum proteins gives results which do not agree completely with those obtained by other methods and that there is no way of determining which is the "correct" result.

Further, the binding power of the protein in the extracts for the bromphenol blue stain is not known, and it may differ from that of albumin. Therefore, the results on the extracts obtained by paper electrophoresis in terms of "protein nitrogen" present are to be regarded as only semiquantitative.

Determination of glycoproteins.—The extracts were analyzed for glycoproteins by paper electrophoresis with periodic acid–Schiff reagent according to a modification of the procedure of Grönwall and Köiw (8, 10). For the glycoprotein determination a larger sample than that used in the bromphenol blue method for serum proteins (0.03 ml. instead of 0.006 ml.) is required, since the stain for the glycoproteins is not so effective, especially in view of the fact that the background acquires a deep pink color against which the purple color of the glycoproteins must be seen. A higher current than that used in the serum protein paper electrophoresis procedure is also recommended (5 milliamperes instead of 2.5 milliamperes).

The glycoprotein paper electrophoresis procedure did not prove completely satisfactory. For bands too light to be recorded by the Analytrol there is no satisfactory method of elution for quantitative evaluation of color with the spectrophotometer. Neither the Sohar staining procedure nor the Drevon and Donikan procedure (11) recommended for glycoproteins gave improved results.

To establish standards for comparison with the glycoproteins of the extracts, bovine glycoprotein (Fraction VI) (Nutritional Biochemicals Co.) and normal bovine serum were used. With a 0.2% glycoprotein solution, which would be expected to give very strongly stained glycoprotein bands, only one light band was found, at approximately 50 to 60 mm. from the application point toward the anode.

With normal bovine serum, three definite glycoprotein bands, with centers at 30, 65, and 115 mm. from the application point toward the anode, are present. If the papers were stained with bromphenol blue (as in the usual serum protein procedure) instead of with periodic acid–Schiff reagent, three bands with positions approximately at the positions of the glycoprotein bands were obtained. This coincidence of glycoprotein and of serum protein bands indicates that the glycoproteins of normal bovine serum are not separated from some of the serum proteins on paper electrophoresis under

the conditions of the glycoprotein procedure; indeed, the glycoprotein bands found in normal bovine serum are undoubtedly due, at least in part, to the globulin fractions, which are known to contain carbohydrate.

Since none of the extracts gave glycoprotein bands sufficiently colored to be evaluated quantitatively on the Analytrol, and since elution of the bands is not satisfactory, the evaluation of the amount of glycoprotein present in a band is based only on the depth of color of the band and is qualitative only.

EXPERIMENTAL RESULTS

Steerhide corium.

Chemical analyses.—The results of the analyses for total nitrogen of the extracts (before dialysis) are given in Table I. There is a steady decrease in the amount of total nitrogen in the extracts with successive NaCl solutions.

TABLE I

TOTAL N CONTENT OF EXTRACTS OF STEERHIDE CORIUM

Extract	Extraction Period (hr.)	Total Mg. N per 100 Ml. Extract (before dialysis)	Total N Extracted as % of Total Corium N		Total Mg. N per 100 Ml. Dialyzed Extract	Protein N as % Total Corium N	
I—5% NaCl	4	19.0	0.83		15.0	0.66	
II— "	18	11.3	0.49	1.72	9.7	0.42	1.43
III— "	4	6.9	0.30		6.1	0.27	
IV— "	2.5	2.2	0.10		1.9	0.08	
V—0.1*M* Na_2HPO_4	17	4.0	0.17		3.8	0.16	
VI— "	4	2.4	0.10	0.32	2.2	0.10	0.29
VII— "	2.5	1.1	0.05		0.8	0.03	
VIII—0.1*M* citrate buffer (pH 3.6)	18	3.2	0.14		——	——	

When the phosphate solution was used for Extract V, however, the amount of total nitrogen increased, which would indicate the extraction of nitrogen-containing material which was not soluble in the NaCl solution. The decrease in nitrogen in the case of the three phosphate extracts may be related to the fact that the second and third extracts were not in contact with the squares so long as the first extract.

The total nitrogen extracted in each extract, calculated as percent of the total nitrogen of the corium samples used, is also given in Table I. The total corium nitrogen was obtained from analysis of the original corium split (6.78% nitrogen). Even after eight extractions the total nitrogen extracted

is quite low (2.18% of the corium nitrogen). The large amount of nitrogen-containing material extracted by the NaCl solutions is undoubtedly related to the presence of soluble nonprotein-nitrogen-containing components (NPN) present in the fresh corium. This type of material would, of course, be extracted by the first extracting solvent used, independent of its nature.

A measure of the contribution of nonprotein-nitrogen-containing compounds to the total nitrogen figures in the extract is given by a comparison of the figures for total nitrogen before and after dialysis against water, since the nonprotein nitrogen (but not the protein nitrogen) will dialyze, and therefore will be largely absent from the extracts after dialysis. As expected, the nonprotein nitrogen is greatest in the earlier extractions and gradually decreases (from approximately 4 mg. per 100 ml. extract to 0.2–0.3 mg.). If it is assumed that the total nitrogen after dialysis is a measure of protein nitrogen, then the protein nitrogen extracted, as percent of the total corium nitrogen, may also be calculated (Table I, Column 6).

The results of the analyses for hydroxyproline carried out on the dialyzed extracts and on the corium split are given in Table II. If the hydroxyproline

TABLE II

HYDROXYPROLINE CONTENT OF DIALYZED EXTRACTS OF STEERHIDE CORIUM

Extract	Hydroxyproline (Microg. per 100 Ml. Dialyzed Extract)	Total Soluble Collagen** (as % of Total Corium Collagen)
I—5% NaCl	563	0.04
II— "	305	0.02
III— "	230	0.02
IV— "	60	0.00
V—0.1*M* Na_2HPO_4	135	0.01
VI— "	135	0.01
VII— "	75	0.00
VII—0.1*M* citrate buffer (pH 3.6)	413*	0.03*

*This value is on the undialyzed (as-is) extract.
**The corium split analyzed 4.44% hydroxyproline.

content of the soluble collagens of the extracts and of the fibrous collagen of the corium split is assumed to be 14%, the amount of collagen can be calculated from the hydroxyproline value.

From the results in Table II it would appear that each of the salt solutions extracts a different type of soluble collagen since the figure is higher for the initial extraction with any one solvent, falls as the extraction progresses with

TABLE III

PAPER ELECTROPHORETIC EXAMINATION OF CONCENTRATED EXTRACTS OF STEERHIDE CORIUM

Extract	% of Total N Lost on Concentration	Total Mg. Protein N per 100 Ml. Concentrated Extract (from Paper Electrophoresis)	% of Protein N of Concentrated Extract Found on Paper Electrophoresis	Positions of Glycoprotein Bands (Mm. from Application Point)
I—5% NaCl	21	43	39	28 mm. toward cathode
II— "	39	27	51	28, 44, and 55 mm. toward cathode
III— "	52	12	48	31, 44, and 56 mm. toward cathode
IV— "	45	2	22	32, 46, and 59 mm. toward cathode
V—0.1*M* Na_2HPO_4	12	14	42	band at application point
VI— "	apparent increase	12	35	band at application point
VII— "	0	0	0	no bands
VIII—0.1*M* citrate buffer (pH 3.6)	31	6	—	6 mm. toward cathode, and at application point

that solvent, and then rises again when a different salt solution is used for the extraction. This finding is in agreement with the work of others on the presence of several types of soluble collagen components in hide, each with a distinctive solubility (3). Some of the soluble collagen of Extracts V to VIII, however, may be solubilized fibrous collagen, as indicated by histological examination of the extracted corium (1).

Paper electrophoresis.—Since the corium extracts lost protein during their concentration for the paper electrophoretic examination, it was necessary to reanalyze the concentrated extracts for total nitrogen. From a comparison of these nitrogen figures with the nitrogen figures for the unconcentrated extracts, the percent of the total nitrogen lost on concentration was calculated (Table III). It should be noted that the concentrated extracts probably contain all the nonprotein nitrogen of the original solutions and that the loss of nitrogen is probably confined to protein nitrogen. Therefore, the loss of protein nitrogen would be even greater than the calculated percent total nitrogen loss shown in the table.

The differences in the percent nitrogen loss in the extracts reflect differences in the proteins present in the extracts and also differences in the salting-out action of the various salts.

Paper electrophoresis (in quadruplicate) of each of the extracts by the procedure for serum proteins gave a single bromphenol blue–staining protein band. This band was essentially a widening of the originally applied band and did not show any definite migration either to the anode or to the cathode. Under the conditions of this experiment (2.5 milliamperes, Veronal buffer [pH 8.6, ionic strength 0.075], 16 hour run), normal bovine serum shows one albumin and three globulin bands with bromphenol blue.

It might be thought that the presence of salts or of $Ca(OH)_2$ in the extracts might influence the electrophoretic behavior of the proteins of the extracts, despite the fact that large amounts of buffer are present However, 1:1 mixtures of normal bovine serum and each of the following solutions (5% NaCl, 0.1*M* citrate buffer [pH 3.6], 0.1*M* Na_2HPO_4, half-saturated $Ca(OH)_2$, or 10% $CaCl_2$) give paper electrophoretic patterns identical with that obtained with normal bovine serum alone.

One of the globulin bands of normal bovine serum remains at the application point and does not migrate on application of current. While this globulin band therefore shows behavior on paper electrophoresis under the conditions of the run similar to that of the protein band of the extracts, on the basis of this evidence alone it can only be stated that the protein is similar to the globulin in electrophoretic behavior. This is not definite proof that the protein of the extracts is a globulin.

Elution of each of the bands with 0.5% Na_2CO_3 and determination of the optical density at 590mμ of the eluate gave the results shown in Table III

(Column 3). The protein nitrogen present in each of the concentrated extracts is not directly known; but if the reasonable assumption is made that none of the nonprotein nitrogen was lost in the concentration, the protein nitrogen can be calculated by subtracting from the total nitrogen of the concentrated extracts the value of the nonprotein nitrogen. The values for the protein nitrogen of the concentrated extracts obtained in this way are much higher than those obtained by paper electrophoresis (Table III, Column 4). From these data it is probable that the protein (or proteins) present in the extracts does not stain well with bromphenol blue compared with bovine albumin. It is believed that the amount of dye bound by a protein is largely a measure of the basic groups present. With human serum proteins, albumin probably has greater binding power for bromphenol blue than do the globulins (12).

In the paper electrophoretic examination for glycoproteins of the concentrated Extracts I to VIII, only very light ban dsshowed on the papers above the pink background color after staining with periodic acid–Schiff reagent. These bands were so light that the possibility that they may have been artifacts exists, although they appeared in each of four duplicate runs. The possible positions of the centers of the bands are shown in Table III (Column 5). Contrary to the results with the glycoprotein solution and with normal bovine serum where the bands showed that migration to the anode had occurred, some of these glycoprotein bands appeared on the *cathode* side of the application point.

Whole steerhide.

Histological examination.—Penetration by the extracting solutions might be expected to be less complete with the whole hide than with the corium split, since stirring was not used to facilitate extraction, the whole hide was thicker than the corium splits, and penetration was through grain and corium instead of through two corium surfaces. The extracted hide samples were examined microscopically; the results of the penetration studies are recorded in Table IV (Column 2). It can be seen that, with the exception of citrate, penetration was incomplete with all the solutions. Consequently, the extracts probably do not contain the maximum amounts of proteins which can be obtained from whole hide by four of the extracting solutions.

None of the extracted hide pieces showed any gelling of the collagen fibers.

Chemical analysis.—The results of the chemical analyses of the concentrated hide extracts are given in Table IV. Since the amount of nonprotein nitrogen has been greatly reduced below that of the original solution, the total nitrogen in the concentrated solution may be regarded as essentially a measure of the protein nitrogen. The total mg. nitrogen extracted from 100 g. hide by each of the solutions is also shown in Table IV.

TABLE IV

TOTAL NITROGEN AND HYDROXYPROLINE CONTENTS OF CONCENTRATED EXTRACTS OF WHOLE STEERHIDE

Extract		Penetration of Hide	Total Mg. N per 10 Ml. Concentrated Extract	Total Mg. N Extracted from 100 G. Hide	Hydroxyproline (Microg. per 10 Ml. Concentrated Extract)
5% NaCl-	1	lower 1/4	10.7	50.1	9
	2		2.9		0
	3		3.1		5
Citrate buffer-0.1*M*, pH 3.6	1	complete	10.4	44.7	420
	2		2.8		105
	3		1.7		153
Na_2HPO_4, 0.1*M*	1	lower 1/3	11.9	49.8	7
	2		2.3		0
	3		2.4		0
$Ca(OH)_2$, half-saturated-	1	lower 1/2	15.3	96.9	26
	2		8.0		0
	3		9.0		11
10% $CaCl_2$-	1	lower 1/3	6.9	37.2	80
	2		3.3		87
	3		2.2		214

The order of extraction of total protein nitrogen from whole hide by the extracting solutions used, under conditions in which complete penetration of the hide did not occur except in the case of citrate, is: half-saturated $Ca(OH)_2$ > 5% NaCl or 0.1*M* Na_2HPO_4 > 0.1*M* citrate buffer (pH 3.6) > 10% $CaCl_2$. It should be noted that, while citrate is the only solvent which penetrated the whole hide layer, it ranks second lowest in order of total mg. protein nitrogen extracted.

Since the extractions of the corium split were carried out consecutively with the different extracting solutions, it is not possible to correlate the results of these extractions with those of the whole hide when the extracting solutions were the same, except in the case of the first solvent used (5% NaCl). In this case the corium yields more of the soluble proteins than the whole hide. However, as previously shown, the extraction of the whole hide was not so complete as that of the corium, as only the lower one-fourth of the whole hide appears to have been penetrated by the NaCl solution. More exhaustive extraction of the whole hide would probably give a higher yield of soluble proteins.

TABLE V

PAPER ELECTROPHORETIC EXAMINATION OF CONCENTRATED EXTRACTS OF WHOLE STEERHIDE

Extract		Total Mg. Protein N per 100 Ml. Concentrated Extract (from Paper Electrophoresis)	% of Protein N of Concentrated Extract Found on Paper Electrophoresis	Position of Glycoprotein Bands	Depth of Color of Glycoprotein Bands
5% NaCl-	1	10	9	0	very light
	2	2	7	0	very light
	3	4	13	0	light
Citrate buffer-0.1*M*, pH 3.6	1	6	6	0; 13 mm. toward anode	light; very light
	2	2	7	0	light
	3	3	18	0	very light
Na_2HPO_4, 0.1*M*-	1	21	18	0	medium
	2	4	20	0	very light
	3	7	29	no bands	——
$Ca(OH)_2$, half-saturated	1	46	30	0	very dark
	2	—	—	0	dark
	3	6	7	0; 17 mm. toward anode	dark; light
10% $CaCl_2$	1	14	20	0	medium
	2	0	0	0	light
	3	0	0	0	very light

The results of the analyses of the concentrated extracts for hydroxyproline are given in Table IV (Column 5). The order of extraction of hydroxyproline-containing material (presumably soluble collagens) is: 0.1*M* citrate (pH 3.6) > 10% $CaCl_2$ > half-saturated $Ca(OH)_2$ > 5% NaCl > 0.1*M* Na_2HPO_4. The citrate solution, as expected, is a particularly effective extraction agent for this material.

The much lower figures for hydroxyproline obtained in these extractions as compared with the corium extractions (Table II) is probably due in part to the effect of the week-long freezing of the hide before extraction, and also to the less stringent extraction conditions. Gross, Highberger, and Schmitt (13) have already shown that skin frozen more than a week yields very little salt-soluble collagen.

It can be seen from a comparison of the hydroxyproline content of the extracts with the results of the other analyses (Tables IV and V) that the amount of hydroxyproline-containing material in an extract does not parallel the total nitrogen figure, the glycoprotein, or the protein nitrogen value obtained from paper electrophoresis (bromphenol blue-staining).

Paper electrophoresis.—Paper electrophoresis (in quadruplicate) of each of the extracts by the procedure for serum proteins gave a single bromphenol blue-staining protein band, similar to that obtained with the corium extracts. In the case of the phosphate extracts there was some "tailing" toward the anode. The two last $CaCl_2$ extracts gave bands which were too light to be recorded by the spectrophotometer. As was done with the corium extracts, the "protein" nitrogen found on paper electrophoresis with bromphenol blue dye was compared with the "protein" nitrogen found in the concentrated extracts by Kjeldahl analysis (on the assumption that the non-protein nitrogen was negligible) (Table V, Column 3). With the whole hide extracts an even smaller percent of the protein nitrogen was found on paper electrophoresis; this would indicate differences in the types of protein present in the two types of extracts.

The results of the glycoprotein paper electrophoretic examination of the whole hide extracts is also given in Table V (Columns 4 and 5). It can be seen that, contrary to the results obtained with the hide corium, the whole hide extracts contain appreciable amounts of glycoprotein, even though the penetration of the hide by the extracting solution was incomplete. Some of the glycoprotein bands appeared dark to the eye, but they were still too light to be evaluated quantitatively on the Analytrol. In agreement with the results obtained with normal bovine serum some of the bands of the hide extracts migrated to the anode; but it should be noted that the positions of these bands differ from those of normal bovine serum.

It can be concluded from the qualitative evaluation of the results that the order of extraction of glycoproteins from whole steerhide, under conditions in which complete penetration of the hide did not occur except in the

case of citrate, is: half-saturated $Ca(OH)_2$ > 10% $CaCl_2$ > 0.1*M* Na_2HPO_4 > 5% NaCl and 0.1*M* citrate buffer (pH 3.6). Although citrate is the only solvent which penetrated the whole hide layer, it is one of the poorest solvents for glycoprotein extraction. The effectiveness of the lime solutions as a solvent for glycoproteins is in agreement with its general use for this purpose. When the relatively less thorough conditions of extraction are considered, it must be concluded that the whole hide is a better source of glycoproteins than the corium split.

The best extracting solution of those tried, in terms both of the glycoprotein and the total protein extracted, is half-saturated $Ca(OH)_2$. However, this solution is a very poor extracting medium for hydroxyproline-containing material (presumably soluble collagens).

DISCUSSION

One fact which should be noted in the paper electrophoretic studies with bromphenol blue stain is the absence of the albumin and of two globulin bands of normal bovine serum. Humphrey, Neuberger, and Perkins (14) have shown that rabbit skin contains serum proteins. Cooper (4) has already reported that five electrophoretic components, of which one is presumably serum albumin, are present in NaCl extracts of bovine hide, and the resolving power of the paper electrophoresis method used here is sufficient to pick up the albumin band.

With the whole hide extracts perhaps one factor is that the hide was frozen for a week before extraction, although freezing would not be expected to cause any appreciable denaturation and loss of solubility of serum proteins. However, the corium split had been frozen for no longer than 20 hours before extraction. A second factor which might be of importance is the fact that the extracts were held at −20° C. for periods of time up to two months before examination, and some denaturation might have taken place during this period. In some of the extracts a slight precipitate was present when the solution was thawed, and this material might represent denatured serum proteins. A third factor which may be of importance with the whole hide but not with the corium extracts is a possible loss of serum proteins on concentration.

SUMMARY

Extracts of the corium split of steerhide were made successively with 5% NaCl, 0.1*M* Na_2HPO_4, and 0.1*M* citrate buffer (pH 3.6). Extracts of whole steerhide which had been frozen one week were made with 5% NaCl, 0.1*M* Na_2HPO_4, 0.1*M* citrate buffer (pH 3.6), half-saturated $Ca(OH)_2$, and 10% $CaCl_2$. Except in the case of citrate, penetration of the whole hide by the extracting solvent was not complete. The extracts were analyzed for total nitrogen and hydroxyproline and were examined by paper electrophoresis for serum proteins and glycoproteins.

With each of the extracts only one protein fraction (which is possibly a globulin) was obtained by paper electrophoresis; other serum proteins were absent. Glycoproteins were either absent or present in very small amounts in the corium extracts. Larger amounts of glycoproteins were found in the whole hide extracts. Most of the glycoprotein was present as one electrophoretic fraction which did not correspond to any of the three glycoprotein fractions of normal bovine serum.

With the whole hide the order of extraction of hydroxyproline-containing material (probably soluble collagens) was: 0.1*M* citrate buffer (pH 3.6) > 10% $CaCl_2$ > half-saturated $Ca(OH)_2$ > 5% NaCl > 0.1*M* Na_2HPO_4. The order of extraction of total protein nitrogen was: half-saturated $Ca(OH)_2$ > 5% NaCl or 0.1*M* Na_2HPO_4 > 0.1*M* citrate buffer (pH 3.6) > 10% $CaCl_2$. The order of extraction of glyproteins was: half-saturated $Ca(OH)_2$ > 10% $CaCl_2$ > 0.1*M* Na_2HPO_4 > 5% NaCl and 0.1*M* citrate buffer (pH 3.6).

ACKNOWLEDGMENT

Appreciation is expressed to the staff of the Tanners' Council Laboratory for advice and assistance during the course of this work and to the Ernest Griess Fellowship Fund which made this study possible. The histological examinations and evaluations were carried out by William Roddy and Mrs. Jean Tancous; some of the extracts were prepared with the assistance of William Roddy, Robert Lollar, and Peggy Brueggemann; and many of the analyses were carried out by Peggy Brueggemann.

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Received October 5, 1959.

THE PRACTICAL IDENTIFICATION OF SOME CONDENSED TANNINS IN LEATHER BY CHROMATOGRAPHIC AND PHOTOMETRIC METHODS

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ABSTRACT

Methods are described for the identification of some condensed tannins present in sole, chrome-retan, and "mordanted" upper leathers. The methods are based on the use of conventional paper chromatography coupled with selective spray reagents and on the generation of anthocyanidins from condensed tannins. The anthocyanidins may be recognised by their colorations, by chromatographic procedures (R_F values), and by their light-absorption characteristics. A number of practical examples are cited. Both the usefulness and the limitations of the methods are evaluated.

INTRODUCTION

Recent advances in the chemistry of condensed tannins (1,2) provide novel methods of establishing the identity of some of the vegetable extracts commonly used for leather manufacture. Methods now available still have obvious limitations, and as always, difficulties will be experienced with blends containing more or less equivalent proportions of a number of extracts. Nevertheless, they furnish a means of establishing with greater certainty the nature of "ordinary" and "sulphited" tannins in sole and retan leathers, and also in lightly "mordanted" upper leathers.

In this paper the application of conventional paper chromatography, selective spray reagents, and the leucoanthocyanidin reaction used in conjunction with photometric procedures will be described. The efficiency and limitations of the methods will be considered for each of the leather types cited above.

*Paper read at the 10th Annual Convention of the South African Section of the Society of Leather Trades Chemists in Port Elizabeth on October 16, 1958.

EXPERIMENTAL AND RESULTS

Preparation and extraction of leather samples.

Sole leather.—Conveniently 5–10 g. sole leather is ground to a fine powder in a Wiley mill. The powder is extracted with acetone/water (1:1) for 24 hr. and filtered, and the clear filtrate is concentrated to dryness under vacuum. Flexible sole-leather types usually contain lower percentages tannins than the more rigid conventional sole leathers, but both usually give sufficient solid extract (0.5–1 g.) for further examination. Leathers tanned with extracts suspected to be heavily sulphited may be extracted by increasing the proportion of water in the acetone/water mixture. During evaporation of the extract a white precipitate frequently separates out after removal of the acetone which evaporates first. This should be filtered off immediately, as it consists mainly of soluble protein-like material and fats.

Vegetable retanned leather.—The presence of chrome on collagen fibres increases the affinity of tannins for this substrate. Also, it is unusual for retanned leathers to be heavily loaded with vegetable tannins, as is normal for sole leather. A combination of these factors often limits the extractibility of tannins from vegetable-retanned leathers. Usually, however, sufficient tannins (10–50 mg.) may be extracted from a suitably ground sample (*see* above) of such leather (5–10 g.) Where the tannins cannot be satisfactorily extracted, the dry leather (5 g.) may be ground and treated as for mordanted leather (below).

Lightly mordanted chrome upper leather.—The relatively high proportion of chrome usually completely inhibits the extraction of the low proportion of surface tannins present. The leather is accordingly ground to a powder, and no extraction is attempted.

Paper chromatographic methods.—The extracted tannins (3–10 mg.) are dissolved in 3 to 5 drops methanol or some other solvent which effects complete solubility. The whole solution is now applied as a single spot to a Whatman no. 1 two-dimensional paper chromatographic sheet, and the paper is irrigated with the upper phase of water-saturated sec-butanol (butan-2-ol) or n-butanol (butan-1-ol)—acetic acid/water (4:1:5) for the first direction, and after drying, with 2% acetic acid for the second direction. We have found it convenient to use a sheet 18¼″ x 11¼″ (half the standard 18¼″ x 22½″ size) and to use the short dimension for the first, and the long dimension for the second, direction. Upward irrigation usually occupies overnight (5 P.M. to 8:30 A.M.) for the first, and 6 hr. (9 A.M. to 3 P.M.) for the second, direction. Chromatograms run simultaneously on stainless steel frames enable direct comparison with each other.

Spraying reagents.—Chromatograms developed under the above conditions should have the components spread out in a well-separated and recognisable pattern. The chromatograms are usually colourless in appearance excepting for a weak brown trail leading from the origin (point of application of the tannin mixture). They may be examined under ultraviolet light in a darkened room, when yellow and blue fluorescent spots usually become visible. The former are likely to be due to flavonols, flavones, chalkones, and aurones, and the latter to substances such as coumarins, the cinnamic acids, and their derivatives. Both sulphited and ordinary quebracho extracts are, for example, readily recognisable on two-dimensional chromatograms by a series of brilliantly yellow-green fluorescent components (direction 1, in a straight line from the origin), while wattle tannins contain two such components in much lower concentration.

Following examination under ultraviolet light the chromatograms may be sprayed with one or more of the following reagents:

(i) Diazotised benzidine reagent (Lindstedt [3]) consisting of (*a*) 10% sodium nitrite solution and (*b*) 5 g. benzidine + 14 ml. conc. HCl +980 ml. water. Two parts (*a*) are added to 3 parts (*b*), and the reagent is used immediately. Chromatograms are washed in a dish in running tap water shortly after spraying.

(ii) Vanillin toluene-p-sulphonic acid (Cartwright, Roberts, and Wood [4]). 1 g. vanillin and 0.5 g. toluene-p-sulphonic acid in 25 ml. absolute ethanol. Chromatograms are lightly sprayed and heated at 80°–90°C. for 5–10 min.

(iii) Ammoniacal silver nitrate (Bate-Smith [5]). 3 g. $AgNO_3$ in 10 ml. water treated with 6*N* ammonia until 1 drop redissolves the silver oxide precipitate formed. After spraying, chromatograms must be washed in a dish, first with distilled water followed by 0.1% sodium thiosulphate solution, and finally with tap water.

(iv) Ferric alum reagent. 3% $Fe_2(SO_4)_3(NH_4)_2SO_4$ (Analar) in water used immediately after preparation.

Chromatograms of leather extractives may be run concurrently with those of reference extracts for comparison and may be sprayed with a number of the above reagents if necessary.

Leucoanthocyani(di)n reaction.—The wide-spread distribution of leucoanthocyanins in nature was demonstrated first by the Robinsons (6) and later by Bate-Smith (7–9). Leucoanthocyanins (i.e., glycosides) and leucoanthocyanidins (i.e., aglycones) are compounds which on treatment with HCl under appropriate conditions yield anthocyanidins (pigments responsible for the pink, red, and blue colorations in most flowers). Pigman, Anderson,

Fischer, Buchanan, and Browning (10) in 1953 first showed that typical condensed tannins such as wattle (mimosa), quebracho, and mangrove, and also others yield red colorations which appear to be anthocyanidins on treatment with HCl. The conditions for the generation of the anthocyanidins from the tannins are quite critical due to the tendency of the latter for self-condensation. Pigman *et al.* (10) suitably overcame this difficulty by using alcoholic-HCl solutions under pressure. Roux (11, 12) and Roux and Evelyn (13) identified anthocyanidins formed from wattle and quebracho and also from other tannins. The patterns of anthocyanidins produced differ and are characteristic for most extracts. Their recognition therefore serves as an additional method of identifying the extracts present in leather.

Conditions of generation of anthocyanidins from tannins.—Aqueous 3*N* HCl has been used for the conversion of leucoanthocyanins into anthocyanidins (6–8). Hydrochloric acid is, however, well known for its excellence as a condensing agent in organic chemistry, and in particular in catechin chemistry, as shown by Freudenberg *et al.* (14–17). Polymeric tannins appear to condense more readily than catechins; and in aqueous medium condensation predominates, and few or no anthocyanidins result. In the alcoholic solution, however, the tendency of the tannins towards condensation is reduced, and degradation into anthocyanidins occurs under the conditions of Pigman *et al.* (10), provided the tannins are sufficiently dispersed in solution, i.e., at high dilution, during the reaction.

3.0 mg. tannins are dissolved in a mixture of 3*N* HCl/isopropanol (propan-2-ol) (1:4) in a tube fitted with a ground-glass stopper or closed by a ground-glass joint, the lid being held down by strong springs. The closed tube is heated on a boiling waterbath for 40–90 min., during which period a scarlet coloration develops. The contents of the tube (5 ml.), after cooling, are all streaked onto a narrow strip 3.5″ x 18″ Whatman no. 3 or 3 MM chromatographic paper by means of repeated streaking and drying in a current of cool air. The chromatogram is developed in 90% formic acid/3*N* HCl (1:1) (11) or in water/acetic acid/conc. hydrochloric acid (10:30:3) (7). The R_F values of the various anthocyanidins (I, II, and III) (Fig. 1) are shown in Table I.

Quebracho extract gives mainly one prominent pink band due to fisetinidin chloride (II:R = H) (11–13). Traces of a second red lower R_F band is visible if the streaking is done at exceptionally high concentration. Its presence is not usually noticed. Sulphited quebracho gives the same anthocyanidin, fisetinidin, but in lower yield, presumably due to the bisulphiting treatment.

Mangrove extract is also rich in leucoanthocyanidins and usually yields only one anthocyanidin, cyanidin (II:R = OH), which has a red colour.

Wattle, or mimosa, extract is characterised by three bands visible after chromatography of the products of the leucoanthocyani(di)n reaction. These bands appear to be due to robinetinidin (red), fisetinidin (pink) and a third orange-yellow band of higher R_F (0.60 in 90% formic/3*N* HCl).

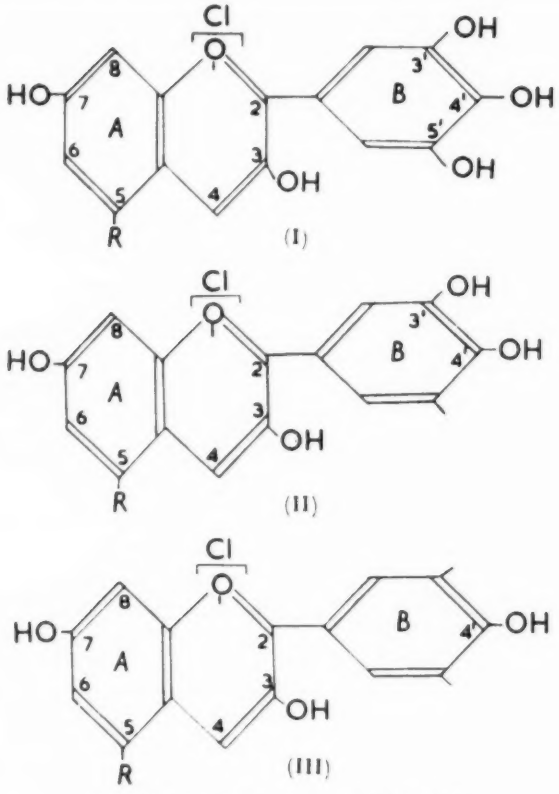

FIGURE 1.—Anthocyanidins I, II, and III.

TABLE I

R_F VALUES OF VARIOUS ANTHOCYANIDINS

Anthocyanidin	Color	R_F Value in Solvent System	
		90% formic acid 3*N* HCl (1:1)	Water/acetic acid/conc. HCl (10:3e:3)
Delphinidin (I:R=OH)	Blue-red	0.11	0.29
Cyanidin (II:R=OH)	Red	0.22	0.50
Pelargonidin (III:R=OH)	Pink	0.33	0.71
Robinetinidin (I:R=H)	Deep red	0.26	0.57
Fisetinidin (II:R=H)	Pink	0.43	0.73
3.7.4′-trihydroxyflavylium chloride (III:R=H)	Orange	0.60	0.95
3.7.8.3′.4′-pentahydroxy-flavylium chloride	Deep red	0.30	0.55

Chestnut, cube gambier, and Burma cutch give no leucoanthocyanidin reaction, but oak bark gives delphinidin (I:R=OH) (18). Tannins derived from *Eucalyptus* spp., e.g., Mallet and Myrtan give high concentrations of anthocyanidins (19), but these have not been studied in detail. Hydrolysable tannins give no anthocyanidins under the appropriate conditions.

Anthocyanidin reference-compounds.—The use of anthocyanidin reference-substances for the comparison of the various shades of red and of the R_F values of the different anthocyanidins generated is often desirable. Pure anthocyanidins may be synthesised from simple starting materials, but synthesis is often difficult and laborious, and a number may be obtained from plant sources by the following method due to Bate-Smith (7).

A number of petals of blue, red, or pink flowers (*see* below) are inserted in a test tube, covered with 3*N* HCl, and heated on a waterbath for 10–15 minutes. The solution assumes the red colour of the flower, and the petals usually decompose into a pulp. The latter is filtered off through glass-wool, and the solution transferred to a very narrow 0.75 x 15 cm. test tube. The anthocyanidin present in aqueous solution may be concentrated by the addition of 3–10 drops n-amyl alcohol followed by shaking of the tube. The amyl alcohol, which preferentially extracts the anthocyanidin, will rise to the surface on standing and may be spotted on the chromatogram as reference substance.

Blue delphiniums furnish delphinidin, scarlet roses give cyanidin, the most common of the naturally occurring anthocyanidins, and pink pelargoniums or geraniums give mainly pelargonidin. Anthocyanidins of the resorcinol series require synthesis, but robinetinidin may be generated from the heartwood extractives of *Robinia pseudacacia* (20), fisetinidin from the heartwoods of *Schinopsis* spp. (quebracho) (21) or *Acacia mollissima* (black wattle) (22), 3.4′.7-trihydroxyflavylium chloride (and fisetinidin) from the heartwoods of *Guibourtia* spp. (23), and 3.7.8.3′.4′-pentahydroxyflavylium chloride from the heartwood of *Acacia melanoxylon* (24). Geissmann and Hinreiner (25) give an excellent index to the presence of various anthocyanidins of the phloroglucinol series in flowers.

Identification of anthocyanidins generated from tannins.—Apart from their R_F values in two solvent systems (Table I) the anthocyanidins may be distinguished by their different colours (Table I) and from other artificial pigments by their conversion into blue pseudobases on fuming the paper with ammonia vapour.

The variation in colour of the anthocyanidins is reflected in their different absorption maxima in the visible range (Table II). These may be determined by running the chromatograms on Whatman no. 1 paper (no. 3 or no. 3 MM is too opaque) and afterwards, after the paper has been aired for 48–72 hr. to allow the escape of HCl, formic, and acetic acids, the coloration is excised in

TABLE II

ABSORPTION MAXIMA OF ANTHOCYANIDINS AND THEIR ALUMINIUM COMPLEXES EXAMINED ON PAPER CHROMATOGRAMS

Anthocyanidin	$\lambda_{max.}$ (mμ)	$\lambda_{max.}$ (mμ) of aluminium complex	$\Delta\lambda$
Delphinidin	555	580	25
Cyanidin	545	560	15
Pelargonidin	530	no complex	
Robinetinidin	532	570	38
Fisetinidin	525	545	20
3.4′.7-Trihydroxy-flavylium chloride	485	no complex	
3.3′.4′.7.8-Pentahydroxy-flavylium chloride	540	590	50

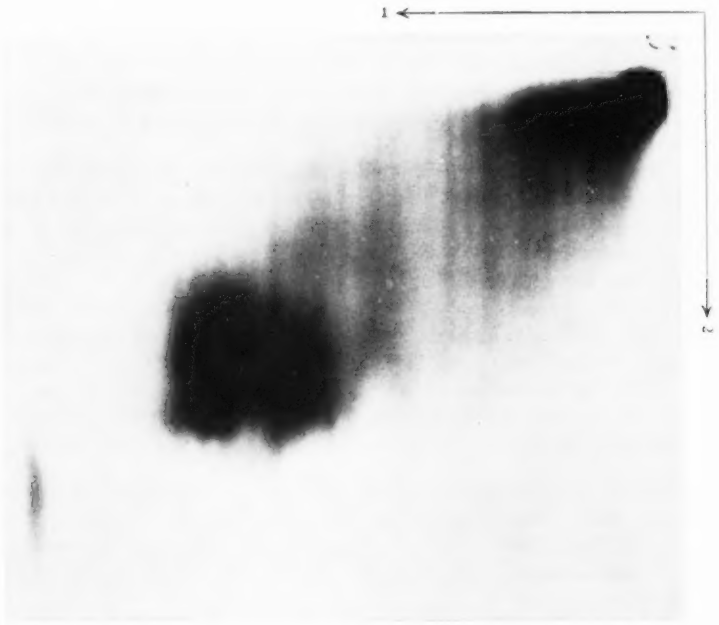

FIGURE 2.—Chromatogram of commercial quebracho ordinary extract. This chromatogram, like those in Figs. 3 and 4, was run by the upwards migration method using n-butanol/acetic acid/water (6:1:2) for direction 1 and 2% aqueous acetic acid for direction 2. The chromatograms measured 16″ x 16″ and were run for 24 hr. in direction 1 and for 14 hr. in direction 2. Chromatograms were sprayed with ammoniacal silver nitrate.

a strip with parallel edges to fit into the cell-carriage of a Beckman (model DU) spectrophotometer. The absorption maximum of each coloration may be determined using the white chromatographic paper as reference-standard according to the method of Bradfield and Flood (26) (Table II).

FIGURE 3.—Chromatogram of commercial chestnut extract.

Where orthohydroxy groups are present in anthocyanidin pigments, they coordinate with aluminium chloride either in solution (*see* Geissman *et al.* [27, 28]) or on paper chromatograms (*see* Roux [12]) to give blue colorations (Table II). The shifts in absorption maxima produced on complexing with aluminium are characteristic of each anthocyanidin.

FIGURE 4.—Chromatogram of commercial mimosa (wattle) extract. The chromatograms of the commercial extract of wattle are never very clear on a photograph, although with the eye the areas of high concentration which stand out clearly on a chromatogram of the fresh-bark extract are readily discernible. However, the chromatograms of wattle, quebracho, and chestnut are easily distinguishable, as is evident from the photographs.

Leucoanthocyanin reaction on lightly mordanted chrome leather.—Upper leathers may contain relatively high proportions of chrome in relation to vegetable tannin, thus making the vegetable tannins almost nonextractable

even with the best solvent systems, due to the enhanced affinity of the chromed leather for vegetable tannins. The tannins may nevertheless be identified under these circumstances by placing 0.2–0.5 g. ground leather sample in the pressure tube and adding the 3*N* HCl/isopropanol (1:4) mixture as before. During the initial stages of heating, the tube is shaken frequently in order to keep the leather particles from congealing. The collagen gradually hydrolises into solution, while the tannins are free to react with the hydrochloric acid in the usual way to give scarlet colorations when leucoanthocyani(di)ns are present. The clear solution obtained is streaked on to Whatman no. 3 paper as before, and the chromatogram is developed.

Interference caused by the presence of dyes and pigments.—Many local and imported upper leathers contain pigments or dyes often consisting of composite colorations in which, amongst others, mauves, reds, and pinks predominate. These dyes have been found, in some instances, to migrate at the same rate as some of the anthocyanidins and may be mistaken for anthocyanidins. Confusion may be avoided by extracting the pigments with acetone/water or methanol/water and subjecting these to chromatography in the solvent system used. This will assist in the interpretation of the final chromatograms obtained after HCl treatment.

Anthocyanidins may always be distinguished by their conversion to blue colorations on fuming with ammonia vapour.

DISCUSSION

For the identification of vegetable tannins present in leather the techniques employed depend on the concentration of tannins in the leather, on whether they have been modified by chemical treatment (namely, by bisulphiting), and on the presence of interfering salts in the leather.

In most sole leathers the fibres are overloaded with vegetable tannins, and even the interfibrillary spaces are usually partly taken up with small amounts of solid extract drummed in from tanning liquors of high concentration. These tannins are not attached to the collagen and are, therefore, readily extracted with methanol, methanol/water, or acetone/water mixtures. The extractives may be examined by two-way paper chromatography, and the pattern of distribution is shown up with the aid of the various spraying reagents recommended in the experimental section. The presence of salts does not normally cause much interference with the migration of the compounds on the paper chromatograms, but where excessive salts are present, they may be eliminated by drying the extract at 105 °C. under vacuum for two hours and by extracting the dried tannins with absolute ethanol or methanol. Should there be doubt regarding the pattern of distribution of components, the tannins may be subjected to treatment with HCl/isopropanol under pressure,

and the nature of the anthocyanidin pigments formed (if any) established and used for confirmation.

Sulphited extracts present a problem in that the typical distribution of tannin components on paper chromatograms is usually altered (29, 30), and a high concentration of product, nonmobile in alcoholic solvent and moving with the solvent front in the water direction, is present. This is presumably the bisulphite-tannin adduct (29). In such instances the bright greenish-yellow fluorescent flavonols present in quebracho, and to a lesser extent in wattle, react either partly or not at all, and they may be recognised easily on two-dimensional chromatograms. Confirmation may also be obtained by the formation of anthocyanidin pigments, where leucoanthocyanidins are present in the original tannins.

For chrome-retan leathers the concentration of vegetable tannins is usually low, and their enhanced affinity for the substrate due to chromed collagen usually makes extraction difficult. Fractional extraction obtained in such instances does not alter the pattern of anthocyanidins formed, although the distribution of tannin components on two-dimensional chromatograms is usually not recognisable.

So high is the affinity of tannins for chromed collagen that where tannins are used for lightly mordanting chromed leather, they appear to be irreversibly fixed. In such instances complete degradation of the collagen by hydrolysis under the conditions used for the leucoanthocyanidin reaction furnishes a clarified solution in which the anthocyanidins formed are often recognisable.

When dealing with problems of identification cognisance should be taken of the fact that of the vegetable tannins, three, namely, quebracho, wattle (mimosa) and chestnut, enjoy by far the biggest consumption (in this order) and widest distribution. Two of these, namely, wattle and quebracho, give easily recognised patterns of anthocyanidins on treatment with alcoholic HCl under appropriate conditions, and their main areas of distribution are well known. Wattle, due to its high solubility, is often used as the basis for tanning blends. Chestnut gives no leucoanthocyanidin reaction and is difficult to identify.

To illustrate the chromatographic methods outlined, the following examples which have been investigated by the author are described.

(*a*) A sample of extremely flexible sole leather of American (U.S.A.) origin, suitable for men's wear, was extracted with acetone/water (1:1). The extract gave a pattern on two-dimensional paper chromatograms typical of a sulphited extract, after spraying with ammoniacal silver nitrate (29, 30). Under ultraviolet light the brilliant yellow-green fluorescent components present were typical of quebracho. On treatment with HCl-isopropanol only fisetinidin (single light pink band) was generated, confirming that the leather was tanned with sulphited quebracho.

(*b*) Chrome-retan upper leathers of British and South African origin were repeatedly extracted with acetone/water (1:1) mixtures, and the solutions were evaporated under vacuum. Only traces of vegetable tannins could be extracted, and the extractives did not give a distribution on two-dimensional chromatograms which could be recognised due to interference. The extracted tannins gave a red solution when subjected to the leucoanthocyanidin reaction, and the chromatographic pattern of the distribution of the pigments (two red and one yellow) was typical of mimosa extract. In one instance (similar sample of unknown origin) a pink dye component interfered with the interpretation of the chromatogram. The dye was pink in colour and migrated to the same position as fisetinidin in the 90% formic 3*N* HCl solvent. Its presence was easily demonstrated by running a chromatogram of the extractives in 90% formic acid/3*N* HCl, before generation of the anthocyanidins. The dye also remained pink on fuming with ammonia vapour, behaviour which is not typical of anthocyanidins (*see* experimental section).

(*c*) Samples of imported upper leathers (British origin) were surface-mordanted and completely resistant to extraction with acetone/water. The powdered leather was inserted in the pressure tube, and a weak but clear scarlet solution was obtained after complete disintegration of the leather in 3*N* HCl/isopropanol. The complete contents of the tube were streaked on to thick paper, and the developed chromatogram showed three faint bands (two red and one orange) typical of mimosa tannins.

Complex blends present difficulties, but where mimosa extract forms the major proportion of the extract, its predominence or presence may be shown by the leucoanthocyanidin method. Mangrove, oak, and some other tannins should also be recognised by the latter method. The use of the photometric methods, although useful, is not essential for the successful application of the chromatographic techniques.

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Received October 12, 1959.

We must give science even greater emphasis than it is now receiving if America and the Free World are to survive the Soviet threat. Leaders in science who bemoan the lack of creativity in their field and stress the need for increasing the number of creative scientists deserve our interest and backing.

But science alone cannot solve all our problems. We must equally encourage the same spirit of pioneering and venture throughout our business structure. It is ironic that while business admires and pleads for creativity and pioneering in science, it increasingly discourages these same qualities in other areas.

In too many firms, business discourages the executive from pioneering and exploring. Instead, business presses him increasingly into a mold of conformity. In the interest of orderly procedure, business more and more encourages, if not orders him, to do as others have done before him. Business too often looks askance at originality and initiative. Businessmen often talk about the need for "new blood," but they generally want it to run to the same blood type. A recent article in *Nation's Business* pinpoints this attitude. It suggested that rugged individualists are unwanted. Business actually wants, the writer says, "creative conformists who are proven profit makers with vision."
—Louis E. Wolfson, *Chairman of the Board*, Merritt-Chapman & Scott Corp.

Therefore, from every standpoint, as businessmen we have an obligation to utilize maximum originality and pioneering in all phases of our day-to-day business operations in order to continue American leadership and a healthy economy. Unfortunately, it has become an increasing habit to describe a good part of our day-to-day operations as "routine business". Nothing in our business world should ever be regarded as completely "routine". The business world must keep pace with the pioneering spirit so dramatically evident in our science and technology. Every new technological advance demands, in fact, a broader range of view by our business executives, whatever their sphere of activity.
—Louis E. Wolfson, *Chairman of the Board*, Merritt-Chapman & Scott Corp.

LIFE LINES

Brief Biographies of Our Contributors

DR. CLARA DEASY holds the Ernest Griess Fellowship and is Associate Professor of Basic Science in Tanning Research at the University of Cincinnati where she completed both her undergraduate and postgraduate work. She has taught at Oberlin College and Temple University Medical School and carried out research on proteins at the University of Illinois and at California Institute of Technology. She has some thirty scientific publications to her credit.

DR D. G. ROUX graduated from the University of South Africa in 1945 with an M. Sc. degree (distinction) for his work on essential oils of rutaceous plants. After a brief period as research chemist for the United Tobacco Company, Cape Town, he joined the staff of the Leather Industries Research Institute, Grahamstown, in 1947, where at present he holds the post of Senior Research Officer. Dr. Roux has specialized in the chemistry of wattle tannins and has published works on tannins, catechins, and anthocyanidins in British, German, and American chemical and leather literature. He received the Donald Burton Prize (1950), the Ph.D. degree of Rhodes University (1952), and the Fellowship of the Royal Institute of Chemistry (1955). During 1954, on a travel grant from the Leather Institute, he studied catechin chemistry with Professor Karl Freudenberg at the Chemical Institute, Heidelberg University, Germany, and chromatography at the Low Temperature Research Station, Cambridge, England.

Recognizing that progress depends on a continuing fund of new discoveries, the chemical industry is now encouraging more basic research in its laboratories. In 1956 the National Science Foundation reported: "The chemical industry far exceeds all others in the magnitude of its basic research program," a conclusion based on a survey of basic research expenditures in 1953 by private industry. Again in 1957 the industry's expenditures for basic research accounted for the largest proportion of company-financed basic research by U. S. industry. . . .The growing storehouse of new knowledge opened up by the chemical industry's fundamental research helps all of science. For many years scientists in the industry have been important contributors of scientific knowledge through the journals and meetings in the fields of physical and natural science. About 40% of the total number of authors listed in the program for the September 1958 meeting of the American Chemical Society were from industry. Almost all chemical companies carrying out fundamental research have a well-defined publications program and a number of the larger organizations publish volumes of their own research. A recent estimate placed an average $26,000 cost behind each technical paper published by a scientist from industry reporting on basic research.

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HERBERT A. TETREAULT, SSUL Laboratory, A. C. Lawrence Leather Company, Peabody, Massachusetts.

PAST PRESIDENTS

G. A. KERR, W. H. TEAS, H. C. REED, J. H. YOCUM, F. H. SMALL, H. T. WILSON, J. H. RUSSELL, F. P. VEITCH, W. K. ALSOP, L. E. LEVI, C. R. OBERFELL, R. W. GRIFFITH, C. C. SMOOT, III, J. S. ROGERS, LLOYD BALDERSTON, J. A. WILSON, R. W. FREY, G. D. MCLAUGHLIN, FRED O'FLAHERTY, A. C. ORTHMANN, H. B. MERRILL, V. J. MLEJNEK, J. H. HIGHBERGER, DEAN WILLIAMS, T. F. OBERLANDER, A. H. WINHEIM, R. M. KOPPENHOEFER, H. G. TURLEY, E. S. FLINN, E. B. THORSTENSEN.

SECRETARY'S NOTICE

The following set of ALCA Journals is available for sale:

1906-1954 Bound volumes
1955-1959 Unbound volumes

Address inquiries or offers to Secretary's Office, American Leather Chemists Association, University of Cincinnati, Cincinnati 21, Ohio.

COUNCIL MEETING MINUTES

February 12, 1960
Sheraton-Plaza Hotel
Boston, Massachusetts

A meeting of the Council was held with President Maeser presiding. Members of the Council present were I. C. Somerville, H. T. Tetreault, E. E. Presley, and C. E. Retzsch. Others present were O. V. Wederbrand, H. G. Turley, and Dominic Meo.

Mr. Maeser reported that Walter Schultz of Elkland Leather Company had found it necessary to resign as Chairman of the Tanning Symposium for the 1960 Annual Meeting and that Jack Wagoner of Armour Leather Company had agreed to become Chairman. This action was unanimously approved by Council.

Mr. Maeser reported that Dr. Windus, Editor of the *Journal* of the ALCA, had agreed to allow *Das Leder* to publish the paper presented by Dr. Francis Schmitt at the meeting of the International Union in Munich. The *Journal* expects to receive a paper for publication on the same subject in the near future. This action was unanimously approved.

The question of a vacancy on the Executive Council of the International Union of Leather Chemists Societies was discussed, and a decision was reached to nominate Dominic Meo. Mr. Maeser will so notify Mr. Forsyth, Secretary, and Dr. Grassmann, President, of the International Union.

Oscar Wederbrand reported on Philadelphia as a possible site of the 1961 meeting of the International Union. Dominic Meo reported on Boston and Washington, D. C., as possible sites. After a long discussion of the advantages and disadvantages of each of the sites and of all possible dates, it was unanimously voted that the meeting should be held in Washington, D. C., on June 25 to June 30 inclusive, 1961.

Council instructed President Maeser to write Dr. Grassmann, President of the International Union, and invite the Union to hold its 1961 meeting in the U.S.A. on the dates and at the location noted above.

Prepared by

M. Maeser

AUDIT REPORT

Arthur W. Holmes
Certified Public Accountant
Cincinnati, Ohio

February 26, 1960

The American Leather Chemists Association, Inc.
The University of Cincinnati
Cincinnati, Ohio

Gentlemen:

In accordance with the request of Dr. Fred O'Flaherty, Secretary-Treasurer, we have examined the balance sheet of The American Leather Chemists Association, Incorporated, as of December 31, 1959, and the related statements of income and surplus for the year then ended. Our examination was made in accordance with generally accepted auditing standards, and accordingly included such tests of the accounting records and such other auditing procedures as we considered necessary in the circumstances.

The report of the examination is presented herewith, together with the following exhibits:

Exhibit I: Balance Sheet at December 31, 1959, and comparison with December 31, 1958.

Exhibit II: Statement of Income for the Year Ended December 31, 1959, and comparison with the year ended December 31, 1958.

Exhibit III: Statement of Surplus for the Year Ended December 31, 1959, and comparison with the year ended December 31, 1958.

Exhibit IV: Investment securities owned at December 31, 1959.

The commercial account balance on deposit with the Central Trust Company was confirmed independently and was reconciled with the cash account of the Association.

Accounts receivable were not circularized; this procedure was considered unnecessary in view of the small balance of each account, and in view of the fact that of the total accounts receivable of $725.30 at December 31, 1959, approximately eighty-five per cent of the total was collected in January, 1960. By other auditing procedures, we have satisfied ourselves of the accuracy, validity, and collectibility of the accounts.

The investment securities, Exhibit IV, were confirmed by Dr. Fred O'Flaherty. In addition, we examined the bonds in detail. All bonds are in the safe deposit box at the Central Trust Company.

In 1959, there were no bond purchases or sales.

Accumulated interest on the Series F bonds has been added to the original cost of $3,700.00 par value of Series F bonds owned at December 31, 1959, At December 31, 1959, the total accumulated value of the Series F bonds is $4,810.00, of which $1,110.00 represents accumulated interest. In the year 1959, all bond investments earned interest of $1,465.00.

Dues received in advance totaling $230.33, advertising received in advance, totaling $27.75, and subscriptions received in advance totaling $3,448.87 were verified in detail and were reconciled with the controlling accounts.

Net income for the year ended December 31, 1959, was $1,200.79, compared with $2,056.38 for the year ended December 31, 1959, or a decrease of $855.59. The causes of the decreased net income are analyzed in Exhibit II. In the year 1959, revenues increased $1,407.72, and expenses increased $2,263.31. The net income is satisfactory, but it is not in accordance with the modern financial trend. It is recommended that dues to members of the Association be increased, because of the increased cost of printing of the Journal, and that advertising and subscription prices (to non-members) be increased. The present trend is not sound financially. In addition, secretarial and editorial salaries have not been increased for several years.

In our opinion the accompanying balance sheet and related statements of income and surplus present fairly the financial position of The American Leather Chemists Association, Incorporated, at December 31, 1959, and the results of its operations for the year then ended, in conformity with generally accepted principles of accounting, applied on a basis consistent with that of the preceding year.

Respectfully submitted,

ARTHUR W. HOLMES
Certified Public Accountant

EXHIBIT I

BALANCE SHEET AT DECEMBER 31, 1959 AND COMPARISON WITH DECEMBER 31, 1958

	December 31, 1959	December 31, 1958	Increase or Decrease* 1959 over 1958
ASSETS			
Current Assets			
Cash on hand	$ 2.12	$ 3.11	$ .99*
Cash in bank	7,670.86	7,658.61	12.25
Accounts receivable, advertisers	600.85	1,260.03	659.18*
Accounts receivable, reprints	124.45	76.00	48.45
Accounts receivable, Directory	–0–	37.00	37.00*
Total Current Assets	$ 8,398.28	$ 9,034.75	$ 636.47*
Investments			
U. S. Savings Bonds, series F (cost $3,700.00 plus $1,110.00 accrued interest at December 31, 1959	$ 4,810.00	$ 4,645.00	$ 165.00
U. S. Treasury Bonds, 2.5%; 1972-1967, at cost	33,207.50	33,207.50	–0–
U. S. Treasury Bonds, 2.5%; 1968-1963, at cost	8,842.50	8,842.50	–0–
U. S. Treasury Bonds, 2.25%; 1962-1959, at cost	10,000.00	10,000.00	–0–
Total Investments	$56,860.00	$56,695.00	$ 165.00
Total Assets	$65,258.28	$65,729.75	$ 471.47*
LIABILITIES AND CAPITAL			
Current Liabilities			
Accounts payable	$ 80.08	$ 112.26	$ 32.18*
Federal income tax withheld	211.02	237.40	26.38*
City income tax withheld	13.01	14.99	1.98*
Advertising received in advance	27.75	57.75	30.00*
Dues received in advance	230.33	214.50	15.83
Subscriptions received in advance	3,448.87	3,046.42	402.45
Total Liabilities	$ 4,011.06	$ 3,683.32	$ 327.74
Capital			
Earned surplus (Exhibit III)	$60,247.22	$60,046.43	$ 200.79
Reserve for foreign meetings	1,000.00	2,000.00	1,000.00*
Total Capital	$61,247.22	$62,046.43	$ 799.21*
Total Liabilities and Capital	$65,258.28	$65,729.75	$ 471.47*

EXHIBIT II

STATEMENT OF INCOME
FOR THE YEARS ENDED DECEMBER 31, 1959 AND DECEMBER 31, 1958

	December 31, 1959	December 31, 1958	Increase or Decrease* 1959 over 1958
Revenues, General			
Dues, including *Journal* subscriptions	$11,174.00	$11,377.00	$ 203.00*
By-laws and Methods booklet	93.40	64.20	29.20
Directory	850.12	789.66	60.46
Interest income	1,465.00	1,361.46	103.54
Total General Revenues	$13,582.52	$13,592.32	$ 9.80*
Revenues, *Journal*			
Advertising	$14,057.92	$13,676.15	$ 381.77
Subscriptions	6,026.31	6,158.76	132.45*
Journal sales	849.58	886.41	36.83*
Reprints	2,163.32	1,142.29	1,021.03
Dues, ISLTC	747.50	563.50	184.00
Total *Journal Revenues*	$23,844.63	$22,427.11	$1,417.52
Total Revenues	$37,427.15	$36,019.43	$1,407.72
Expenses, General			
Salary of Secretary Office	$ 4,600.00	$ 4,600.00	–0–
Office operating expense	739.73	1,197.33	$ 457.60*
Uncollectible accounts expense	358.00	430.00	72.00*
Directory expense	1,064.49	962.68	101.81
By-laws and Methods	674.20	613.80	60.40
Annual meeting expense	176.64	483.20	306.56*
Council meeting expense	77.00	117.83	40.83*
Auditing	200.00	200.00	–0–
Leather Chemists Union dues	120.09	119.74	.35
Total General Expenses	$ 8,010.15	$ 8,724.58	$ 714.43*
Expenses, *Journal*			
Printing	$21,038.28	$19,103.45	$1,934.83
Managing editor, compensation	2,499.96	2,500.00	.04*
Managing editor, expense	359.84	223.01	136.83
Editor-in-chief, compensation	600.00	600.00	–0–
Editor-in-chief, expense	146.56	194.67	48.11*
Miscellaneous expense	22.00	60.78	38.78*
Storage	350.00	350.00	–0–
Insurance	60.00	62.00	2.00*
Copyright registration	48.00	48.00	–0–
Reprints	2,237.10	1,282.41	954.69

	December 31, 1959	December 31, 1958	Increase or Decrease* 1959 over 1958
Exchange charges	6.18	4.55	1.63
Translations and abstracts	390.04	439.47	49.43*
Committee expense	10.00	107.69	97.69*
Advertising written off	300.60	111.00	189.60
Advertising discounts allowed	147.65	151.44	3.79*
Total *Journal* Expenses	$28,216.21	$25,238.47	$2,977.74
Total Expenses	$36,226.36	$33,963.05	$2,263.31
Net Income	$ 1,200.79	$ 2,056.38	$ 855.59*
Net income distributed: General	$ 5,572.37	$ 4,867.74	$ 704.63
Journal	(4,371.58)	(2,811.36)	1,560.22
Total	$ 1,200.79	$ 2,056.38	$ 855.59*

EXHIBIT III

STATEMENT OF EARNED SURPLUS
FOR THE YEARS ENDED DECEMBER 31, 1959 AND DECEMBER 31, 1958

	Year Ended December 31, 1959	Year Ended December 31, 1958	Increase or Decrease* 1959 over 1958
Balance, January 1	$60,046.43	$58,990.05	$1,056.38
Add Net Income			
General	5,572.37	4,867.74	704.63
	$65,618.80	$63,857.79	$1,761.01
Deduct Net Loss			
Journal	4,371.58	2,811.36	1,560.22*
	$61,247.22	$61,046.43	$ 200.79
Deduct			
Allocation to reserve for foreign meetings	1,000.00	1,000.00	–0–
Balance, December 31	$60,247.22	$60,046.43	$ 200.79

LEATHER EXPERTS AT LTI——Dr. Harold G. Turley, extreme left, former president of the American Leather Chemists Association and recently president of the International Leather Chemists Association, is appearing as visiting lecturer at Lowell Technological Institute in this spring semester under the auspices of Rohm & Haas Co., Philadelphia, with which he is affiliated as research associate. Dr. Turley is presenting three two-hour lectures in a two-day span each month through May. Shown with him, left to right, are Dr. Albert E. Chouinard, head of LTI's Department of Leather Chemistry; Dr. Edward F. Mellon, head of Composition Investigations, Hides and Leather Laboratory, U. S. Dept. of Agriculture, Agriculture Research Service, Eastern Utilization Research and Development Division, Philadelphia, Pa.; and Dorrance H. Goodwin, executive director, LTI Research Foundation. Dr. Mellon has been at LTI to discuss the USDA-LTI Research Foundation project, "Investigations on the isolation and identification of compounds comprising the basement membrane of cattlehides for use in the development of improved unhairing methods to yield better quality leather."

DIRECTORY CHANGES

NEW MEMBERS

Chas. Allen, Garden State Tanning, Inc., Fleetwood, Pennsylvania.

Donald E. Finegold, 31 Raymond Circle, Peabody, Massachusetts. Kirstein Leather Company 77 Walnut Street, Peabody, Massachusetts.

Robert H. Fletcher, Technical Service Department, Clinton Corn Processing Company, Clinton, Iowa.

Eric Johnston, Robson Leather Company Limited, Oshawa, Ontario, Canada.

Edwin J. Kaine, 17 Hamilton Road, Woburn, Massachusetts.

Roger J. Langlais, Endicott Johnson Corporation, Upper Leather Processing, Endicott, New York.

P. C. Moreau, 40 Forrest Avenue, Amityville, New York. Corn Products Sales Company, 350 Fifth Avenue, New York 1, New York.

Henry O. Muenzner, A. C. Lawrence Leather Company, Division of Swift and Company, Ashland, Kentucky.

Donald H. Smith, 7651 Salem Street, Lynnfield, Massachusetts. Apex Chemical Company, Inc., 200 S. First Street, Elizabethport 1, New Jersey.

William M. Springer, 15 Rambling Lane, Malvern, Pennsylvania. Surpass Leather Company, c/o J. R. Evans Leather, Second & Erie Avenues, Camden, New Jersey.

Jean J. Tancous, 3060 Ramona Avenue, Cincinnati 11, Ohio. Tanners' Council Laboratory, University of Cincinnati, Cincinnati 21, Ohio.

Carlos A. Tosi, P. O. Box 128, Tomas Ordones No. 60, Cuenca, Ecuador.

CHANGES OF ADDRESS

William F. Allen, A. E. Staley Mfg. Company, 2200 Eldorado Street, Decatur, Illinois.

Alberto Alonso, Calle 10, No. 1307, Caibarian L.V., Cuba.

Fred E. Artz, Howes Leather Company, Columbus, Indiana.

Harvey Austin, 11 Oxford Street West, London, Ont., Canada.

Robert Banner, Diamond Alkali Company, Research Center, P. O. Box 348, Painesville, Ohio.

Thomas J. Barnet, B. D. Eisendrath Tanning Company, Racine, Wisconsin.

Robert E. Barnwell, Robeson Process Company, Box 960, Erie, Pennsylvania.

Jose Behar, Phila Pike, Box 234, RD No. 3, Reading, Pennsylvania.

William C. Beier, 402 North East Street, Coudersport, Pennsylvania.

Robert Biedermann, J. R. Geigy S.A., Schwarzwaldallee 215, Basle, Switzerland.

Thomas C. Blair, Indian Lane, Hackettstown, New Jersey.

Paul E. Breer, Iroquois Oil, Color & Chemical Corporation, Edward Street, P. O. Box 86, Amsterdam, New York.

Charles Cares, c/o Garden State Tanning, Inc., Fleetwood, Pennsylvania.

William P. Cooke, E. I. duPont de Nemours & Company, Industrial & Biochemicals Dept., Wilmington 98, Delaware.

Harold P. Davis, Davis and Son, Ltd., P. O. Box 37, Kingston, Ont., Canada.

Kenneth Folger, 8 Mill Bend Road, Levittown, Pennsylvania.

Stanley Friedland, Triangle Tanning Company, 1820 Webster, Chicago, Illinois.

Alfred Freudmann, P. O. Box 193, Caracas, Venezuela.

Car' F. Good, Lackawanna Leather Company, Hackettstown, New Jersey.

Carlton C. Gordon, Albany Felt Company, P. O. Box 1109, Albany 1, New York.

H. L. Hartman, C. W. Marsh Company, 1385 Hudson Street, Muskegon, Michigan.

William B. Hartnett, 1560 N. Prospect Avenue, Milwaukee 2, Wisconsin.

A. W. Hopton, 561 Creek Road, Lewiston, New York.

Lt. Col. Howard James, Seventh U. S. Army, APO 46, New York, New York.

Frank Jaslowich, 8 Range Heights Terrace, Lynn, Massachusetts.

James M. Kelly, R. M. Phelps Street, Gloversville, New York.

Thomas A. Klein, 11 Brentwood Avenue, Gloversville, New York.

Charles K. Knisely, 391 Delaware Avenue, Oakmont, Pennsylvania. Thompson and Company, 1085 Allegheny Avenue, Oakmont, Pennsylvania.

Merrill R. Leach, Research Department—Brooklyn Lab., Socony-Mobil Oil Company, 412 Greenpoint Avenue, Brooklyn 22, New York.

Stanley Lipowski, 25 Ashwood Drive, Livingston, New Jersey.

Clarence Long, Dyes Department, American Cyanimid Company, 3505 N. Kimball Avenue, Chicago 18, Illinois.

John A. Lowell, 1613 Pine Street, Philadelphia, Pennsylvania. Rohm and Haas Company, 5000 Richmond Street, Philadelphia, Pennsylvania.

Charles A. Martin, E. Hubschman and Sons, Inc., Division of Gera Corporation, 415 N. Fourth Street, Philadelphia 23, Pennsylvania.

Robert E. Merritt, Apt. No. 11, 2121 N. High Street, Columbus 1, Ohio. Chemical Abstracts Service, The Ohio State University, Columbus 10, Ohio.

Harold R. Miller, Jr., 4445 N. Ardmore Avenue, Shorewood 11, Wisconsin.

Guy H. Moberg, 107 Rhame Avenue, Red Wing, Minnesota.

Donald J. Moser, Geo. Moser Leather Company, P. O. Box 249, New Albany, Indiana.

Gabriel M. Oliensis, 1809 N. 22nd, Sheboygan, Wisconsin. Armour Leather Company, Sheboygan, Wisconsin.

Tom S. Perrin, Research Center, Diamond Alkali Company, P. O. Box 348, Painesville, Ohio.

John H. Pierce, Diamond Alkali Company, Research Center, P. O. Box 348, Painesville, Ohio.

Cecil Powell, Tanners' Hide Bureau, 221 N. LaSalle Street, Chicago 1, Illinois.

Robert Putnam, E. F. Drew & Company, Inc., 416 Division Street, Boonton, New Jersey.

Reinhold J. Quatsch, A. Davis & Son, Ltd., P. O. Box 37, Kingston, Ontario, Canada.

Colin D. Quinn, 3528 W. Southland Drive, Franklin, Wisconsin.

John W. Quinn, Jr., K. J. Quinn & Company, Inc., 195 Canal Street, Malden 48, Massachusetts.

John H. Reck, International Shoe Company, 1509 Washington Avenue, St. Louis 66, Missouri.

Kenneth H. Schlifske, 7437 North 51st Blvd., Milwaukee 23, Wisconsin.

Philip J. Shivell, Slip-Not Belting Corporation, P. O. Box 89, Kingsport, Tennessee.

Ralph W. Smitka, Flagg Tanning Corporation, 2116 E. Estes Street, Milwaukee 7, Wisconsin.

George L. Somer, Armour Leather Company, Sole Leather Division, 500 Arch Street, Williamsport, Pennsylvania.

Edward G. Suffern, Suffern Chemical Company, Box 132, Johnstown, New York.

John F. Wagoner, Armour Leather Company, Sole Leather Division, 500 Arch Street, Williamsport, Pennsylvania.

John Wesley Wolfe, Box 400, Acton, Ontario, Canada.

RESIGNATIONS

George E. Best, Manufacturing Chemists' Association, Inc., 1825 Connecticut Avenue, N.W., Washington 9, D. C.

Leroy A. Kramer, Victor Chemical Works, 155 North Wacker Drive, Chicago 6, Illinois.

D. F. Lord, 243 Green Street, Marblehead, Massachusetts.

Leslie A. Maynard, A. C. Lawrence Leather Company, Division of Swift and Company, Ashland, Kentucky.

Raymond A. Mills, Endicott Johnson Corporation, Endicott, New York.

Henry Schreiber, Apex Chemical Company, 200 S. First Street, Elizabethport 1, New Jersey.

Robert H. Sohn, Patjo Leather Company, 4 Union Street, Peabody, Massachusetts.

Louis Strymish, 15 Indian Ridge Road, Newton Centre 59, Massachusetts.

Raymond L. Truche, Truche Leather Company, 98 Foster Street, Peabody, Massachusetts.

FIFTY-SIXTH ANNUAL MEETING

TENTATIVE PROGRAM

SUNDAY, JUNE 19—WEDNESDAY, JUNE 22

LAKE PLACID CLUB, LAKE PLACID, N. Y.

SUNDAY, JUNE 19

TECHNICAL COMMITTEE MEETINGS

(*Afternoon; times to be arranged by Convention Vice-Chairman*)

Hide Powder Committee ..... W. WINDUS
Raw Stock Evaluation Committee ..... T. C. THORSTENSEN
Mineral Leathers Committee ..... N. C. BENRUD
Tannin Analysis Committee ..... E. L. RAYFIELD
Standards and Specifications Committee ..... C. G. TELANDER
Vegetable Leathers Committee ..... R. M. LOLLAR
Physical and Mechanical Properties Committee ..... E. D. COMPTON
Preparatory and Posttanning Processes Committee ..... W. T. RODDY
Tannery Effluents Committee (stand-by basis) ..... E. B. THORSTENSEN

PRESIDENT'S RECEPTION

(*Evening reception prior to dinner*)

MONDAY, JUNE 20

PRESIDENT'S ADDRESS

Mr. Mieth Maeser has asked various officers and committee chairmen to report on activities of the Association.

JOHN ARTHUR WILSON MEMORIAL LECTURE

In order to bring an eminent guest speaker to the Association Meeting, this Memorial Lecture has been sponsored by the Salem Oil and Grease Company. The first lecture in this series will be delivered by Dr. John H. Highberger, past president of the ALCA and well known to all. His major life interest has been in the elucidation of the structure of collagen, and he will lecture on our present state of knowledge in this field.

TECHNICAL PROGRAM

The Depolymerization of Collagen Fibers. A. Veis, J. Anesey, and J. Cohen (Research Division, Armour and Company).—Native bovine corium collagen was partially solubilized at the shrinkage temperature. The soluble portion was fractionated by an ethanol-sodium chloride coacervation technique. The fractions were characterized by a variety of physical techniques including ultracentrifugation and light scattering. The ultracentrifuge studies clearly showed that the depolymerization of the intact fiber did not yield a gaussian weight distribution of gelatin. Several discrete molecular weight species were obtained. Since the depolymerization was carried out with minimal hydrolysis, the molecular species distributions can be related to the state of aggregation of collagen in the intact fiber. The distinction between a "collagen molecule" and the functional entity "a collagen fiber" is discussed.

Kinetics of the "Heat Gelation" of Native Collagen. J. M. Cassel, L. Mandelkern, and D. E. Roberts (Leather Section, National Bureau of Standards).—Collagen molecules can exist at low temperature in dilute solution as highly asymetric, internally ordered structures. Depending on environmental conditions (pH, ionic strength) transformation to the random coil state can be effected by raising the temperature. However, under certain conditions a "gel" is formed on heating, a process which, according to Gross, preserves the integrity and native structure of the molecules. The kinetics of this latter process has been studied from pH 4.5 to 10, over a wide range of temperatures and concentration. Very strong negative temperature coefficients are observed, indicating that the process is nucleation controlled. In the process of "heat gelation" it is suggested that the ordering of the individual collagen molecules is occurring.

GOLF TOURNAMENT

The annual golf tournament is scheduled for Monday afternoon, weather permitting. In case of rain, the Tuesday afternoon technical program will be presented on Monday, and the golf will be played on Tuesday.

ENTERTAINMENT

(*Professional entertainment and dancing in the evening*)

TUESDAY, JUNE 21

TECHNICAL PROGRAM

The Influence of Type of Oil, Degree of Sulfation, and Neutralization Level of Fatty Acids on Some Physical Properties of Side Upper Leather. Conrad L. Ornes, Tanner's Council Research Laboratory.—A laboratory study of some fatliquoring variables applied to "average sides" of side upper leather has indicated that the temper and feel of leather are significantly influenced by the kind of oil and the degree of sulfation of the oil used in fatliquoring. Methods used to determine such characteristics and data to substantiate the findings will be presented. Also it will be shown that the depth of penetration of the oil and the "break" of the leather are not measurably influenced by such factors.

Studies on the Combined Application of Different Types of Tannins. F. F. Miller, Badische Anilin & Soda Fabrik.—The high quality requirement for leather set by the shoe industry on the one hand and the demands of fashion on the other compel the leather manufacturer to find new production methods. These may take the form of a variation in the tanning methods, utilizing the available raw materials and possible means of processing.

Vegetable retanning of chrome leather is at present the most widely practised manufacturing process of shoe upper leather. The introduction of this combination tannage brought about a series of problems, as the prerequisites for vegetable tanning of chrome leather differ greatly from those for tanning pelts.

The paper gives a report on the investigations of the electric charge relationships in the retanning of chrome leather. Based on these relations, methods have been developed for the combined use of different types of tannins. These methods have proved beneficial for the appearance, elasticity, and firmness of the grain as well as for the fullness and feel of the leather.

The combined use of different types of tannins was carried out as follows:

(*a*) Application of synthetic tannins in chrome tanning
(*b*) Application of synthetic tannins in neutralizing of chrome leather
(*c*) Application of synthetic tannins in conjunction with mineral tannins in retanning
(*d*) Application of resin tannins in conjunction with synthetic tannins

The evaluation of the results shows that improved leather properties are obtained by the combined use of different types of tannins.

The Effects of Splitting upon the Physical Characteristics of Leather. Joseph Orietas, Albert Trostel and Sons Co.—Splitting has been an important operation in the manufacture of side leather, and tanners know

from experience the consequences and ill effects of oversplitting upon the physical strength and quality of their leather. The emphasis upon splitting has been further accentuated in recent years because of the increased availability of heavier hides and the frequently arising necessity of using such hides for the production of leather that would have been made out of lighter-weight hides.

The work we are reporting is of practical nature. It was carried out for the purpose of studying and evaluating the effects of splitting upon the strength of our leather under production tannery conditions.

In our efforts to reach a better understanding of the effects of splitting upon the physical strength of leather, we have arrived at some conclusions which have helped us to deal with splitting in a more realistic manner and with greater confidence.

Preliminary Studies of the Dynamic Mechanical Properties of Leather. L. P. Witnauer and W. E. Palm, Eastern Regional Research Laboratory.—The vibrating-reed technique has been applied to the measurement of the dynamic Young's Modulus of elasticity and mechanical loss factor of various leathers. In this test method a specimen of leather about 10 cm. in length and 1 cm. in width is clamped at one end in a vibrator. The frequency of vibration of the reed-shaped specimen is varied, and the amplitude of the free end is measured. A plot of amplitude versus frequency is utilized to determine the resonant frequency and band width from which the dynamic properties are calculated. The effect of such variables as amplitude, frequency, moisture content, and compression on the dynamic behavior of various commercial leathers will be discussed.

Improvement of Some of the Properties of Sole Leathers by Impregnation with Polymers. Joseph R. Kanagy, Leather Section, National Bureau of Standards.—A review is presented of the work on the improvement of the wear of sole leather by impregnation with polymers. Emphasis is placed on the characteristics of the leather which promote ease of treatment and produce the greatest improvements in results. Methods of evaluation of the treated leathers are discussed critically, and a mechanism for abrasion resistance improvement of leather is suggested.

Stratigraphic Distribution of Carbohydrate, Hexosamine, and Hydroxyproline in Cattlehides. Edward F. Mellon and Samuel J. Viola, Eastern Regional Research Laboratory.—Earlier studies have shown that the grain region of cattlehides contains only about one half the concentration of dry matter and nitrogen contained in the corium or central portion of the hide. Several explanations can be hypothesized. One postulates the presence

of mucopolysaccharide gels which hold considerable quantities of water and thus decrease the apparent concentration of dry matter. To test this hypothesis a stratigraphic analysis for components of the mucopolysaccharides was undertaken. A freezing microtome was used to cut consecutive slices of equal thickness parallel to the grain surface of the hide. These slices were analyzed for reducing sugars, hexosamines, hydroxyproline, nitrogen, and total nonfat solids.

When calculated on a dry weight or nitrogen basis, both reducing sugars and glucosamine appear concentrated in the grain and flesh regions of the hide. However, on the basis of a unit volume of hide, there is a smaller amount of both the reducing sugars and glucosamine in the grain region than in the corium. Therefore, the mucopolysaccharides are not likely to be responsible for causing a higher hydration of the grain area.

The hydroxyproline determinations show that a significant part of the protein present in the grain area is not collagen. Thus, the presence of highly hydrated proteinaceous gels could be a possible explanation for the enhanced hydration of the grain area.

A Study of a *Clostridium* Bacterium that Causes Severe Hide Damage. Jean J. Tancous, Tanners' Council Research Laboratory.—A bacterium taken from salt-cured hide samples having severe grain damage has been isolated, and its identity is still being studied. This bacterium is able to tolerate up to 8% salt, and its resistant spore is able to lie dormant in dirty salt and in hide even after liming. It causes severe damage to hide samples as they exist in a pack and can completely digest corium collagen samples in test tube trials. The enzyme that this bacterium produces has been isolated and is comparable to the collagenase of *Clostridium histolyticum* in its digestion of native collagen. Slides and data will be presented.

Elastase Activity of Some Enzymes as Related to their Depilatory Action. T. C. Cordon, A. L. Everett, H. Jones, W. Windus, and J. Naghski (Eastern Regional Research Laboratory).—The fibrous protein elastin, which is usually associated with collagen in animal organs and tissues, has been suggested as an anchoring material for the epidermis of the skin. To obtain data on this concept and a better understanding of the mechanism of enzymatic unhairing, studies were made on the relationship between hair-loosening ability and elastase activity of depilatory enzymes. Elastase activity of depilatory enzymes was measured by three methods. Isolated ligament elastin and formol titration were used in one method for measuring the extent of the action. Intact hide was used in the other two methods. The degree of action on the hide was measured histologically in one case and by a gravimetric procedure in the other. The results obtained with these methods and their comparison with the depilatory activity of the enzymes will be discussed.

Chemical Unhairing. T. C. Thorstensen.—A review is given of previous work on chemical unhairing systems, with particular emphasis on mild alkaline treatments. Using this as a basis, a new fast unhairing system has been developed without the use of lime. Soaked hides are placed in a bath containing relatively large amounts of reducing agents at moderate pH values. This bath works on the soft keratins in the skin, breaking some of the cystine linkages. The hair is not damaged at these low pH values. The first bath is drained, and the hides are rinsed and then placed in a mild alkaline bath containing ammonia. Hair loosening results, and the stock can be unhaired. The system results in controlled swelling and hair saving.

Zirconium Determination by Gravimetric Methods. J. Wendkos and I. C. Somerville, Rohm and Haas Research Laboratory.—A simple method for determination of Zr in tanning materials and in leather was reported by the authors some years ago and is being investigated at present by members of the mineral tannage committee of the ALCA. In the course of preparing solutions of known composition to serve as standards for comparison some new information was obtained, and this is now reported.

Increased utilization of Zr and its salts, especially in connection with the Atomic Energy program, has lead to the publication of many new analytical methods—titrimetric, gravimetric, spectrophotometric—for its determination. Some of the gravimetric methods have now been examined, particularly one based on the use of tartrazine as precipitant. This has led to an improvement in the procedure, and clarified the conditions necessary, for consistent results. The results obtained in this way are compared with those given by some of the standard gravimetric methods.

BANQUET

The guest speaker will be Harold Caldwell Kessinger, who will talk on the "Sunny Side of Main Street". Judge Kessinger has been an editor, publisher, world traveler, and civic leader as well as a New Jersey jurist for three terms.

ENTERTAINMENT

Golf prizes will be presented in a special ceremony following the banquet. Dancing for those who desire will also follow the banquet.

WEDNESDAY, JUNE 22

TANNERS' SYMPOSIUM

Vegetable Tanning of Sheepskin Leather. R. N. Jones, A. C. Lawrence Leather Co.—Vegetable-tanned sheepskin leathers are manufactured today for shoe and boot linings, hatbands, bookbindings, and various specialty

uses such as ladies' and men's wallets, diploma cases, etc. The filling and firming characteristics of vegetable tanning materials lend themselves well to the production of these—the so-called firm-end of sheepskin leathers.

The sheepskin tanner must make allowance for conditions different from those of the tanners of heavy leathers. These include the loose, open-fiber structure of the skins, the variation in acid, salt, and grease contents of pickle-skins from different sources, and for economy reasons, the need of a short process time employing drum tannage. Since the tanner has little control over the beamhouse operations his rawstock has been subjected to, including the pickle, an examination will first be made of the steps necessary for the proper conditioning of the skins prior to receiving the tan. Extracts used will be discussed. Quebracho and wattle are used in large amounts; limited amounts of chestnut and sumac are sometimes employed to modify blends of the above.

The need for minimizing the amount of physical pounding wool-type sheep-skins are subjected to is an important consideration. In this regard, mention will be made of the different practices of feeding the tan liquor and the resultant effects of concentration, rates of penetration, and tannin fixation as they are related to producing quality leathers.

The Scientific Principles of Vegetable Tanning. A. N. Kay, Howes Leather Company.—While the preservation of raw hides and skins by the action of vegetable tanning materials is a comparatively simple matter, the practice of leathermaking is exceedingly complex. Not only must the raw stock be converted into an imputrescible material, but that material must have imparted to it many other properties to make it suitable for the particular end use to which it is to be put. Then, and only then, can the product be considered leather.

Since it is a protein, the properties and behavior of the raw stock are determined by its environmental history, and this must always be kept in mind in any consideration of the factors influencing any particular phase of the over-all process.

Insofar as the actual tannage is concerned, the major factors to be considered are: type or types of tanning material used, time, temperature, concentration, viscosity, degree of dispersion, type and amount of acidity present, pH, neutral salts, and the solvent used. These are briefly discussed not only in terms of current usage but with an eye to the future.

Vegetable Tanning of Bag, Case, and Strap Leathers. C. A. Lauder, Wickett & Craig, Ltd.—Before we consider tanning bag, case, and strap leathers, it is necessary to know what qualities we want in the leather. It must be somewhat firm, yet there must be ample flexibility with no grain

cracks. The condition of the fiber structure in the grain is important. The degree of tannage should equal 60°–65°. Color should be a light pink-tan shade.

Hemlock originally produced this shade, but hemlock stocks have diminished through the years. In succession we have added quebracho, myrobalans, wattle, valonia, and European chestnut. The reason for these additions will be explained. Our blend must contain fermentable sugars as found in hemlock extracts.

Temperature, acidity, and strength of liquors are closely controlled. Methods and materials will be mentioned. The period of tanning time has been shortened from two months to three weeks.

We have experimented for some time with drum and paddle tanning but have not been able to produce high-quality leathers which meet the standards we require. However, we are still trying and shall continue to do so. For we must struggle against increasing competition from other fields. Bag, case, and strap leathers are fashioned into luggage, portfolios, bookbinding, and wallets; they are also used for carving and tooling.

Vegetable Tanning of Upholstery Leather. E. E. Presley, Eagle Ottawa Leather Co.—The art of making upholstery leather is rapidly becoming a science. With modern laboratory equipment and new tanning materials, plus the ever increasing knowledge of the make-up of collagen and its reactance with these new, as well as the old, materials, a better product is the end result. Upholstery leather must have a round feel and be very supple, with good fiber strength and only moderate stretch. Water solubles must be low with a high degree of tannage to absolutely insure a minimum migration of tannin which could cause yellowing of white and pastel finishes.

Some of the above physical characteristics are effected by previous processes in the beamhouse, but the role that vegetable tanning plays is very important. Proper solution concentrations, pH, and temperature, as well as blend of vegetable materials must be accurately controlled. Unable to obtain hemlock bark economically, we substituted Orotan as a replacement tannin to give us the strength and color required by our standards.

Lime split and chrome-retanned upholstery leathers can be produced faster, but vat-tanned straight vegetable upholstery still requires 2½ to 3 weeks tanning time. The role that solvent vegetable tanning will play in the future is visible in the crystal gazing ball, but that is another story.

Vegetable Tanning of Sole Leather. J. F. Wagoner, Armour Leather Company.—The vegetable tanning of sole leather has undergone a number of changes dictated by today's requirements for shoe bottom materials, the availability of tanning materials, and the economic aspects of the industry.

Today the emphasis is on flexible light-weight sole leathers. Therefore, the tanner has reappraised his tanning procedures in the light of existing scientific knowledge and modified his processes so as to be able better to control these characteristics. The time required to tan sole leather has been greatly reduced in the past few years. This has necessitated the installation of much stricter chemical controls. A more judicious selection and handling of tanning materials has resulted in a better utilization of tannin and has had a favorable effect on costs.

The use of pretanning agents, such as aldehydes, lignosulfonates, and syntans, with the subsequent vegetable tanning carried out in rockers or in drums is growing in the industry and may well influence the processing of sole leather in the future.

EXHIBITS

Commercial exhibits to be shown during the Meeting have been arranged by Oscar Wederbrand, Ways and Means Chairman.

CONVENTION COMMITTEE

ROBERT STUBBINGS, Chairman
FRED KLIMPL, Vice-Chairman
HAROLD R. MILLER—Entertainment and Publicity
FRANK CHADWICK—Golf
MRS. THOMAS C. THORSTENSEN—Ladies

ABSTRACTS

Hygienic Properties of "Purified" and "Pure" Toluenes. M. Zeman. *Kozarstvi*, **9**, 122–23 (1959).—Determinations of benzene (by infrared spectroscopy) and thiophene in "purified" toluene (minimum boiling point 100°C., 90% distilling to 120°C.) and "pure" toluene (95% distilling from 109.5° to 111.0°C.) are reported. Only the latter is safe to use for cementing in shoe factories. L.M.

Polymerizates for the Finishing of Leather. V. Pektor and J. Ondracek. *Kozarstvi*, **9**, 109–10, 140–41, 173–75, 203–7 (1959).—A review. Analytical methods for testing leather finishes are described, and results of qualitative and quantitative analyses are given. The first finish coat for corrected grain shoe upper leather should contain only polymerizate dispersions and aniline dyes. Further details of finishing processes are given. L.M.

Complexometric Determination of Sulfate Ions in Syntans. Z. Hrabel. *Kozarstvi*, **9**, 111–12 (1959).—The importance of the determination of SO_4 in syntans and the disadvantages of older methods are discussed. Prepare 0.001*M* Complexon III by dissolving 0.744 g. in 2 l., and standardize it against $PbCl_2$ with Eriochrome Black as indicator. One ml. of 0.001*M* Complexon III equals 0.2072 mg. Pb. Prepare 0.002*N* $BaCl_2$, and standardize it against the Complexon solution. Prepare a buffer solution by diluting 8.28 g. NH_4Cl and 113 ml. of NH_4OH (25%) to 900 ml. To 10 ml. of this buffer add 10 ml. of a 10% solution of $MgCl_2$, dilute it to 50 ml., and titrate it with a 10% solution of Complexon III and Eriochrome Black to a clear blue color. Add this titrated solution to the 900 ml. of buffer, and make to 1000 ml. Finally prepare a 2% solution of *p*-toluidine in ethanol denatured with ether. Make up a solution of the syntan to contain 1 to 1.5 mg. solids per ml. To 5 ml. of the syntan solution add 25 ml. denatured ethanol, 5 ml. of the *p*-toluidine solution, and 5 ml. $BaCl_2$ solution, boil, cool, add the indicator and 5 ml. of the buffer, and titrate quickly to a pure blue color (green in the presence of dihydroxy phenols). The average error of the determination is 0.09% for phenol sulfoacids, 0.69% in pyrocatechol sulfoacids, and 1.28% in 2-naphthol sulfoacids. In commercial German and Swiss syntans, SO_4 contents (dry basis) were: Tanigan Extra B, 6.70%; Extra Special PL, 5.57%; Extra A, 4.59%; Extra Que, 4.69%; Irgatan RBL, 15.43%; HO, 8.24%; Corolan F, 26.76%; Pellutan EZE, 5.23%. L.M.

Auto-oxidation of Vegetable Oils in Tanneries. G. Vágó, J. Reti, and P. Varga. *Kozarstvi*, **9**, 41–44 (1959).—Auto-oxidation of soya bean oil, used in Hungary for stuffing, caused damage to shoe upper leather stored in large piles. Atmospheric O_2 forms a biradical on double bonds of the oil, which may be stabilized as a hydroperoxide, -R-O-OH, or react with a second double bond. Heat is liberated by the reactions. The hydroperoxide can be decomposed by metallic salts. Spontaneous heating of leather depends on the type of oil, its peroxide no., Fe content of the leather, and the volume of the leather piles. The Mackay test has been used to assay oils. The peroxide no. is the equivalent of I_2 liberated by 1000 g. oil from HI solution, expressed as ml. of *N* $Na_2S_2O_3$. A raw soya bean oil had a peroxide no. of 47 and a Mackay test of 210 min. After being stored in glass for 6 mo., the peroxide no. was 136. The Mackay test was lowered to 71 min. by 0.01 g. $FeCl_3$ in 14 g. oil, and to 35 min. by 0.1 g. The Fe in leather may lower the Mackay test. Cr salts have no such effect, and Al only a very small one. Self-heating of leather can be prevented by antioxidants, including vegetable tannins, 0.2% hydroquinone, Na_2S, and Na_2S_4, or by partial sulfation of the oil, which blocks most of the unsaturated bonds. The Mackay test of soya oil was raised from 35 to 74 min. by sulfation with only 2% commercial H_2SO_4 for 3 hr. at 30°–40°C. Epoxy compounds are formed by decomposition of hydroperoxides, and they change further to hydroxy acids. The Japanese white tannage may possibly be explained by the tanning action of epoxy compounds. These tan at pH values above 6, which may account for the fact that chamois tanning is not complete until the leather is treated with Na_2CO_3 solution. L.M.

Theory and Practice of Leather Finishing with Pigment Finishes in the USSR. V. I. Eliseeva. *Kozarstvi*, **9**, 102–03 (1959).—The behavior of pigment-finished leathers was studied by wear tests of experimental shoes.

Apparatuses for determining flex resistance, abrasion resistance, heat stability, and mechanical properties of films are described. Fresh nitrocellulose (**NC**) and polymethacrylate (**PMA**) films stand 780 and 9330 flexes respectively; aged films withstand 250 and 5400 flexes; at 12°C. **PMA** stands only 20 flexes. Durability (% of cracks) was 34 for **NC** and 81 for **PMA**. Films of **PMA** alone are more durable than with a surface coat of **NC**. Of the films tested, polyethacrylate (**PEA**) was most extensible, 2000%. Extensibilities of other films were: **PMA**, 1000%; polyamide AR-669 (**PAR**), 145%; Eukanol binder L (**EL**), 1075%; Eukanol binder W (**EW**), 1340%; linseed oil varnish (**LO**), 48%. Resistances to heat, to flexing at 20°C., and to flexing at –15°C., in that order, were: **NC**, 150°, 787, 141; **PMA**, 80°, >100,000, 57; **PEA**, 100°, >100,000, 300; **PAR**, 120°, 5030, 2000; **EL** and **EW**, 75°–73°, >100,000, 200; LO, 150°, 41,200, 70. A good film should possess low internal friction at –20° and –30°C. and should show a minimum melting point of 90°C. and in some cases 150°C. Best results were obtained with films from methyl and butyl acrylate copolymers containing 5% acrylic acid (**MBA**), or with 1–2% of monoalkylamide of methacrylic acid (**MBM**), and for patent leather, polyurethanes from toluylene diisocyanate and castor oil (**PUC**). Resistances of these films to heat, and percent extensibilities at 20°C. and at –20°C., were: **MBA**, 170°, 1195, 510; **MBM**, 80°–90°, 1920, 65; **PUC**, 140° (decomposes), 200, 0. Resistances to flexing at 20°C. were >100,000, and at –20°, 2000. Resin dispersions penetrate better into leather if they possess small-sized particles, good resistance to salting-out, and low surface tension. The most important factor is the potential difference between charged resin particles and leather fibers. The pH of a dispersion of negatively charged particles should be higher than the isoelectric point of the leather, if maximum penetration is wanted. Formulations made in accordance with the above principles are given. The first coat should be applied to wet leather after setting. Use of **NC** films is being restricted. For patent leather, made almost entirely on goatskins in the USSR, the first coat is made from **NC** and non-drying glyptal resin. The top coat is made from **LO** cooked with Prussian blue and Pb salts at 270°–270°C. Such patent leathers generally stand 1000–3000 flexes. **PUC** has recently been introduced on a large scale for the top coat over the **NC**-glyptal resin coat; it is dried at 60°C. The flexing resistance of such leather is >100,000. L.M.

Dependence of Leather Dyeing on Type of Dyestuff and Preparation of Leather for Dyeing. V. Pektor and J. Ondracek. *Veda a Vyzcum v Prumyslu Kozedelnem*, **5**, 73–100 (1958).—Leathers of a wide variety of tannages and retannages were dyed at pH values of 3.0, 4.5, and 6.0 with each of four different dyes (Chrome Leather Brown 3GN, Alizarine Chrome Red G for Leather, Koregal Blue GG and Koranil Brown ER). Results obtained are reported for each dye, with regards to levelness, intensity, penetration, exhaustion, effect of electrolytes, and fastness to water, fatliquor, and light. L.M.

Determination of Reducing Sugars in Vegetable Tanning Extracts. A. Blazej and M. Mladek. *Kozarstvi*, **9**, 131–34 (1959).—Reducing sugar is measured by determining Cu_2O reduced by the sugar from Fehling's solution. The solutions are first detannized by basic Pb acetate, which, however, also precipitates phenolic nontannins, polyuronides, and polysaccharides. Part of

the reducing sugar is occluded by the voluminous precipitate. The excess Pb must be removed by Na_2SO_4, NaH_2PO_4, or $K_2C_2O_4$. The amount of Cu_2O formed is not strictly proportional to the reducing sugar present, but other methods are no better in this respect. When reducing sugars were determined by the Pb precipitation method in chestnut extract solutions to which 0 to 10 mg. of glucose had been added, 0.00 to 1.04 mg. of glucose was occluded by the precipitate. Similar determinations on spruce extract solutions containing 0.00, 9.4, 18.8, and 28.2 mg. glucose showed occlusions of 0.0, 2.42, 3.63, and 5.6 mg. respectively. Several other tannin-precipitating agents were studied, and best results were obtained with Cu acetate. The precipitate formed is less voluminous and showed practically no occlusions of sugars from chestnut solutions and only 0.20 to 0.36 mg. occlusion with spruce extracts. Filtration is more rapid, and it is not necessary to remove the surplus Cu acetate. To replace the titration of precipitated Cu_2O by $KMnO_4$, which is tedious and not very precise, a complexometric determination of Cu is proposed. When solutions containing from 1 to 25 mg. glucose were analyzed, the differences by the complexometric method were −0.02 to +0.05 mg., and in the $KMnO_4$ method they were −0.13 to +0.16 mg. Murexide is used as the complexometric indicator. For amounts of glucose up to 25 mg. in 50 ml., Cu_2O precipitated is a linear function of the glucose content, and the following factor can be used: 1 ml. 0.001*M* Complexon III = 0.32 mg. glucose. A new complexometric indicator, recommended by Wehber, based on the reaction of ninhydrin with an amino acid, works best in the Cu_2O determination in the ratio of 1 ninhydrin to 5 β-alanine in 50 water. The solution is unstable, and it is better to homogenize a dry mixture of the components. The presence of NaCl makes the color change indefinite. The optimum pH lies between 5.2 and 6.0. *Procedure:* To 25 ml. of analytical tannin solution and 25 ml. water add 25 ml. of a 1.5% solution of Cu acetate. Digest the dark brown precipitate on the water bath at 75°–85°C. for 30 min.; sedimentation is more rapid at the higher temperature. Filter through dry paper, transfer the precipitate to the paper, let drain, and wash to a total volume of 100 ml. filtrate. Pipet 10 ml. each of Fehling's solution A and B into a tall-form beaker, add 50 ml. of the detannized, filtered solution and 3 glass beads, bring to a boil in 3 min., and boil exactly 2 min. Filter the precipitated Cu_2O on a glass filter, and dissolve it in 20 ml. *N* HCl. Dilute the solution to about 100 ml., and neutralize with dilute NH_3 to about pH 8.0. Add 0.1–0.2 g. indicator (murexide), and titrate with Complexon III from yellow to violet. One ml. Complexon III solution corresponds to 0.6354 mg. Cu or 0.32 mg. glucose. Results of determinations of reducing sugars by the usual method and the method here given, expressed as mg. glucose per 25 ml. analytical tannin solution, were: chestnut, 6.96, 7.30; spruce, 14.76, 20.68; wattle, 6.49, 8.01; sumac, 6.99, 7.89; valonea, 5.86, 7.31; urunday, 1.31, 2.56; quebracho, 0.58, 1.36. The differences are due to occlusion of sugar by the Pb-tannin precipitate. L.M.

Corrected Grain Side Upper Leather in Hungary. G. Vajda. *Kozarstvi*, **9**, 10–14 (1959).—In 1953 only 1.4% of all side upper leather was corrected grain; in 1957, 54%. The thickness of casein finish on full grain leather is 0.005–0.020 mm.; on corrected grain, 0.02–0.06 mm. Beamhouse processes have been changed in going from full grain to corrected leather. The process now is: soak 48 hr. at 12°–18°C. in 2 waters; lime in drum with 170–200% liquor, or in paddle with 300–400%, 8.5–10 g. Na_2S (60%) and 10–15 g.

CaO per l., at 20°–23°C. for 18–24 hr. Drums are turned 5 min. every second hr. at 1 rpm. No difference between drum and paddle liming has been noted. The skins are washed 20–30 min. at 25°–30°C., then treated with 0.3–0.5% $(NH_4)_2SO_4$ and 0.5–0.8% lactic acid or HCl. The temperature is raised to 37°C. in 20 min., when 2/3 of the pelt thickness is neutral to phenolphthalein. Then the bate (equivalent to 0.25–0.5% Propen OR) is added and drumming continued for 20–50 min. Pickle in 100% water, 8% NaCl, and 0.8–1.0% H_2SO_4 for 2 hr., then pretan 2 hr. in 70% used Cr liquor (3–5 g. Cr_2O_3 per l.). Tan with 2% Cr_2O_3 in molasses-reduced liquor, 42% basic, half added in 30% float and the remainder after 1 hr. in 40% float. Add 0.3–0.5% Na_2CO_3 in 10% liquor slowly after 2 hr., and drum 2 hr. longer. Next day, add 50% water, drum 1 hr., and horse 24–48 hr. Neutralize with 1% syntan (*o*-cresol sulfonic acid condensed with HCHO and adjusted to pH 5.2 with NH_3) and 0.5–1.0% $NaHCO_3$ in 200% float at 35°C. Wash before and after neutralizing. The surface pH should be 5.5, and the center, 4.8–5.0. Deep shades are drum-dyed; light shades, brush-dyed. Fatliquoring is done in 200% float at 50°–55°C. with 1.0–1.5% neatsfoot oil, 1.2–1.5% sulfated fish oil (100% fat), 0.2–0.3% soap, and 0.1–0.2% ammonia. Retanning follows in 200% liquor at 30°, pH 4.0–6.0, with 2.2–3.8% tannin composed of a mixture of 35–40% of the same syntan and the rest quebracho, wattle, or chestnut extracts. Finishing is done with Eukanol pigments. Smooth plating is done with a rubber plate, 70 hardness, on a felt backing. L.M.

New Methods of Dyeing Furs. R. Vrbacky. *Veda a Vyzcym v Prumyslu Kozedelnem*, **5**, 113–22 (1958).—Dyeing with a fixed volume of liquor on the dry weight, rather than per skin, is advocated; 2000% liquor on the dry weight is suggested. Rabbit skins normally are tanned, fatliquored, dried, sorted, wet back, and dyed. Dyeing without drying out saves labor and steam, but sorting and repairing is easier if done before dyeing. Fatliquoring must precede dyeing. Conditions for "killing" (alkaline pretreatment to make the hair receptive to the dye), mordanting, and dyeing white rabbit skins with oxidation dyes were studied. In killing, no amount of Na_2CO_3 is sufficient unless 2–5 ml. H_2O_2 (30%) is added. For bleaching natural color rabbit skins 5–10 ml. H_2O_2 is necessary, or 20–30 ml. for "intensive" bleaching. Optimum killing was obtained with 5 g. Na_2CO_3 and 0.5 g. Syntapon L (an anionic detergent) per l. for 2 hr. at 35°C. A nonionic detergent is preferable. Studies of mordanting included variations of $K_2Cr_2O_7$ and acetic acid content as they affect exhaustion of dichromate, final pH of mordant bath, and subsequent uptake of dye. Best exhaustion of dye bath (*p*-phenylene diamine) was obtained when the final pH of the dichromate bath was 4.85, but then the bristle hairs are a little lighter. A low pH in the dichromate bath can be compensated by a higher pH in the dye bath. Optimum mordanting was attained with 2 g. $K_2Cr_2O_7$ and 0.5 ml. acetic acid (90%) per l. for 18 hr. at 30°C. Rate of exhaustion of *p*-phenylene-diamine-H_2O_2 dye bath was studied at different dye concentrations. Exhaustion equilibrium is attained more rapidly at higher concentrations, but the degree of exhaustion is lower. Exhaustion equilibrium is attained in 3 hr., but maximum color intensity is not reached even in 24 hr. It makes no difference whether the H_2O_2 is added all at once or in several feeds. The rate of exhaustion depends on the particular dye. In full-scale tests, paddle vats holding 2400 l. were used, and dyeing was done with 2.8 to 5.8 g. dye per l. at an initial temperature of 35°C. If killing, mordanting, and

dyeing are done in one paddle, a false bottom is required to permit intermediate washing. Continuous running of the paddle during dyeing gives more rapid exhaustion, but no greater ultimate exhaustion; it does produce more level shades. Use of oxidation dyes is objectionable on hygienic grounds. Ursatine dyes (IG and BASF) were introduced to replace Ursols because of the instability of the latter, but Ursatines will not color bristle hairs. The merits of various lines of German and Swiss dyes are discussed. L.M.

Czechoslovak Experience with the Chrome-Vegetable Tanning of Sole Leather in Comparison with Other Countries. I. Introduction. M. Spicka and V. Hrabovsky. *Kozarstvi*, **9**, 6–9, 14 (1959).—The literature is reviewed (61 references). The possibility of using a larger percentage of replacement syntans in Cr-vegetable, as compared to straight vegetable tannage, is stressed. Chroming with 0.4–0.7% Cr_2O_3 on pelt weight is recommended. Chrome-vegetable tannage is used exclusively for tanning sole leather in the USSR; it has also been used recently in Hungary, where even shoulders and bellies of good quality are made. In East Germany, sole leather tannage is done with 0.4% Cr_2O_3 in a sugar-reduced liquor of 42% basicity on well-delimed pelt. Neutralization is done with 0.4% Na_2CO_3 followed by 10% Pellutan EZE (a replacement syntan). Vegetable tanning follows in 10 suspenders, 1 or 2 layers, and 3 days in a drum, using 50% each of syntan and vegetable tannins. **II. Experimental.** *Ibid.*, **9**, 135–41 (1959).—The paper of Kucka and Minarik (abstr. *JALCA*, **54**, 166 [1959]) is criticized. In new experiments one bend was tanned by straight vegetable tannage, and the corresponding bend by a Cr-vegetable tannage. The experimental bends were washed, delimed with NH_4Cl or $(NH_4)_2SO_4$, pickled with NaCl and HCl, and tanned overnight with 6–9% Cr alum, neutralized, and piled. Vegetable retannage was done in 3 suspenders and 2 drums, with a total tanning time of 10 days in earlier, and 5 days in later, experiments. The leathers were loaded with sugar, Epsom salt, oxalic acid, sulfated oil, and 4% of Syntan K_2D, shaved, struck out, oiled off, dried, dampened, and rolled. Tannage was done with a blend containing 0% to 60% of replacement syntan; the rest of the mixture was composed of spruce, sulfited quebracho, wattle, chestnut, and oak wood extracts (2 2/3:1:1:1:1). Results of analyses and physical tests are given. The Cr-vegetable leathers contained about 0.9% Cr_2O_3; their fixed tannin contents were nearly the same as for straight vegetable leathers. The prechroming of the bends permitted the use of more syntan in the mixture without sacrificing quality; on the contrary the quality of the Cr-vegetable leathers is equal to or better than that of straight vegetable sole. This was shown by abrasion and wear tests. L.M.

Curing Today. M. Dempsey. *J. Soc. Leather Trades Chemists*, **43**, 434–45 (1959).—The special curing problems of India and Africa are discussed. The use of antiseptics in salt curing was studied by application of orthophenylphenol and pentachlorophenol in resalting market hides for vegetable sole and chrome calf and in curing calfskins. Both orthophenylphenol and pentachlorophenol checked red heat and were undetectable after normal liming. Their use incurred small colored spots on raw skins which were removed during subsequent processing. A combination of soda and naphthalene is shown to give a good cure, resulting in some upgrading of split hides but no difference in leather yield. Curing by aceton dehydration and by gamma radiation is also briefly discussed. J.M.C.

Studies on the Enzyme Depilation of Hides and Skins. I. Depilation Tests with Aerobic Bacteria and Selection of Effective Strains. H. Toyoda and A. Futami. *Repts. Govt. Chem. Ind. Research Inst., Tokyo*, **54**, 343–53 (1959) (English summary, p.xxxviii). **II. Bacteriological Characteristics of Selected Strains.** *Ibid.*, 354–62 (English summary p. xxxiv); *see* abstracts, *JALCA*, **53**, 225 (1958). H.B.M.

Recent Trends in Leather Research in the United States. H. Merrill. *J. Soc. Leather Trades Chemists*, **43**, 431–34 (1959).—A summary of the high lights of leather research in industrial and Government laboratories in the United States. J.M.C.

Use of Sodium Carbonate and Naphthalene in Salting Hides and Their Effect on the Quality of Sole and Upholstery Leather Therefrom. M. Dempsey and R. Elliot. *J. Soc. Leather Trades Chemists*, **43**, 445–51 (1959).—One hundred steerhides for sole leather and 100 cowhides for split upholstery leather were randomized into control (NaCl-cured) and soda-naphthalene-NaCl curing lots. Hides were removed from the cure pile after 3, 6, and 12 months. Examination of finished bends from hides stored for 6 months showed no consistent differences in fiber structure as a result of the difference in cure history. The keeping qualities of the hide were definitely improved by use of soda-naphthalene. The leather from the soda-naphthalene–treated hides stored for 12 months showed some evidence of slightly finer splitting-up and slightly more compact weave pattern than the control. The use of soda-naphthalene had no adverse effect on leather yield. J.M.C.

Experiments on Wool from Copper-Deficient Sheep. R. Burley and F. Horden. *Nature*, **184**, 1725–26 (1959).—Copper deficiency in sheep results in weak wool with abnormal dyeing properties. Such wool is shown, after mild oxidation, to contain a lesser amount of an acid-soluble protein of high S content than normal wool. C.L.D.

Effect of Soap on the Diffusion of Water through Isolated Human Epidermis. F. Bettley and E. Donoghue. *Nature*, **185**, 17–20 (1960).—The diffusion of water through isolated human epidermis is greatly increased by addition of soap to the water and somewhat increased by detergent (a secondary alkyl sulfate). Bile salts do not show a similar effect; decreased surface tension is therefore not the cause of the diffusion effect. The permeability of the epidermis in contact with soap solution returns to normal in a few days when the soap is removed. C.L.D.

Regeneration of Protein from Insoluble Protein-Tannin Compounds. W. Mejbaum-Katzenellenbogen, W. Dobryszycka, J. Boguslawska-Jaworska, and B. Morawiecka. *Nature*, **184**, 1799–1800 (1959).—Protein-tannin compounds of a number of different proteins, including gelatin and proteins of normal biological fluids, were investigated. The proteins could be recovered from the compounds in 90% yield by addition of caffeine. The regenerated proteins did not appear to differ in physical or chemical properties from the original proteins. C.L.D.

Optical Studies. I. Relation of Colour to the Apparent Soilability of Textile Materials. H. Mann and T. Morton. *J. Soc. Dyers Colourists*, **75**, 522–33 (1959).—A study of soiled colored starch showed that the degree of apparent soiling is related to the physical concentration of soil and the reflectance spectrum of the original surface. From the spectrum a characteristic empirical quantity, the SMR value, can be computed, which ranks the colored material in order of relative apparent soilability in a series of materials similar in structure but differing in color. The results are generalized to fibrous materials. **II. SMR Values for a Comprehensive Range of Colours.** H. Mann. *Ibid.*, 534–35 (1959).—The SMR values of 78 colors are listed; from these values any of the dyes may be assessed approximately in respect to its relative liability to show soiling. C.L.D.

PATENTS

Waterproofing. Brit. Pat. 822,673. Monsanto Chemicals Ltd. Appl. July 26, 1957.—Porous materials, e.g. textiles, leather, paper, cardboard, masonry, wood and concrete, are rendered water-repellent by treatment with an amylhydrogenosiloxane and a titanium compound capable of hydrolysis to an oxide of titanium. The siloxane and Ti compound can be applied together as a composition (*see* Group IV (a)) or separately in either order, and the compositions may be in the form of organic solvent solutions or aqueous emulsions. Ti compounds specified are tetra-alkyl orthotitanates and condensed alkyl titanates containing alkyl radicals of 1 to 10 C atoms, ethanolamine titanates and reaction products of $TiCl_4$ with ethylene glycol, propane diol and 1:3- and 2:3-butane diols, which may be further reacted with amines, e.g. alkanolamines, as described in Specifications 804,989, [*Group* IV (a)], 804,990 and 812,505. Methyl-, ethyl-, amyl-, phenyl- and methylphenyl-siloxane resins and/or siloxane oils may also be added. In examples, white cotton fabric is immersed in a white spirit solution or an aqueous emulsion of the composition, squeezed and dried at room temperature and finally heated at 160°C. to cure the composition. Specifications 708,821 and 723,989 also are referred to.

Treatment of Leather; Paper Containing Synthetic Polymers; Proofing Leather, Paper Fabrics &c. Brit. Pat. 822,267. Rohm & Haas Co. Appl. April 12, 1956.—Homopolymers and copolymers of compounds of general formula

$$CH_2{=}CHYAN{-}\overset{R^1}{\underset{|}{C}}{-}\overset{X}{\overset{||}{N}}{-}\overset{H}{\underset{|}{C}}H_2OR^2$$

in which Y is O or S; A is phenylene or an alkylene group having 2–4 carbon atoms, of which a chain of at least 2 extend between the adjoining atoms Y and N; R^1 is H, cycloalkyl or a C_1–C_{12} alkyl group, X is O or S, and R^2 is an alkyl group; may be dissolved in a suitable solvent and applied to various materials including fabrics, paper and leather, as coatings, adhesives and impregnants, to impart water-repellancy, and to prevent decay caused by fungi and bacteria. The materials are then dried to convert the polymers to insoluble and infusible condition. In the examples: (a) N-vinyloxyethyl-N^1-methoxy-

methylurea (Y is O, A is—CH_2CH_2—, X is O, R^1 is H and R^2 is CH_3 in the above formula) and water are heated in the presence of dimethyl azoisobutyrate and the resulting polymer is used as a beater additive for improving the wet strength of paper; and (b) a homopolymer of N-methoxymethyl-N^1-vinyloxyethylthiourea (Y is O, A is —CH_2CH_2—, X is S, R^1 is H and R^2 is CH_3 in the above formula) is added to water containing 1% of an ammonium salt of a styrene-maleic copolymer to give a suspension which may be used to treat fabrics, wood and cellulosic materials to prevent decay caused by fungi and bacteria.

Artificial Fur. Brit. Pat. 820,279. Rona, L. Appl. May 16, 1956.—Animal hairs are formed into a layer upon a flat surface, the hairs being at right angles to the surface, and the ends of the hairs adjacent the surface are formed into a felted layer so as to form an artificial fur. Wool clipped from the sheep, wherein the outer parts of the hair are entangled, may be placed on an endless belt provided with convergent side guides which pack the hairs more closely together, the hairs being maintained in a position at right angles to the belt with the assistance of a vacuum below the belt and/or compressed air above. The lower ends of the hairs may be treated with a hot alkaline detergent solution, heated, and acted upon with a reciprocating brush in a series of steps, which may be repeated, causing felting. The free ends may be carbonized by treatment with sulphuric acid and heat and then washed, and the felted ends may be treated with formaldehyde and coated with rubber latex or a synthetic resin. The finished artificial fur may be passed through a carding machine and cutter. A layer of horizontally disposed fibres may be incorporated in the felted layer or a similar layer of fibres may be incorporated between two plastic coatings applied to the felt. As an alternative method, both ends of the hairs may be felted and the product cut along the centre to provide two artificial furs. Wool which has been cleaned may be treated, the outer ends of the hairs being held by adhesive bands of textile material, by a fine metal screen provided with carding hooks or by a sprayed-on adhesive. Pieces of otherwise useless skins may be matched and placed upon a perforated stainless steel plate and the skins removed by chemical or bacteriological methods to produce an artificial fur.

Coated Fabrics. Brit. Pat. 820,541. Dunlop Rubber Co. Ltd., and Glovers (Chemicals) Ltd. Appl. Aug. 25, 1955.—Sheet materials, e.g. fabrics, coated and/or impregnated with a plasticized synthetic resin are coated, to render them anti-static, with a tertiary amine or a quaternary salt thereof containing at least one oxyalkylene group and at least one long-chain aliphatic radical. Alternatively, the sheet material may be coated and/or impregnated with a composition comprising a plasticised synthetic resin and the amine or quaternary salt. The resin may be polyvinyl chloride, polyvinylidene chloride, polyvinyl acetate, and copolymers of vinyl chloride, vinyl acetate, or vinylidene chloride. The plasticiser may be a diphthalic ester of octyl alcohol which contains small quantities of heptyl and nonyl alcohols. The preferred amines have the formula

$$R—N[(R^1O)_mH]_2$$

where R is the long chain aliphatic radical, m is an integer from 1 to 50 and R^1 represents the group $(CH_2)_p$ where p is an integer greater than 1. Preferred

quaternary salts are those obtained by treating the preferred amines with a compound of the formula $R^{11}X$ where R^{11} represents an alkyl, alkenyl, or aryl group and X an acid radical. In the above formula R may be caprylyl, oleyl, lauryl, myristyl, stearyl or cetyl, R^1 ethylene, or propylene, R^{11} methyl, butyl, or cetyl and X chlorine, bromine, or CH_3SO_4. In Example (1) artificial leather upholstery is made by coating a fabric with a composition consisting of polyvinyl chloride, the plasticiser previously specified, and an amine of formula $(C_{17}H_{33})N[(C_2H_4O)_3H]_2$. In Example (2) artificial leather upholstery is made as in Example (1) but the amine is not included in the composition but rubbed subsequently into the surface whilst in Example (3) a conveyer belt is made by impregnating a fabric with a composition comprising polyvinyl chloride, the plasticiser previously specified and an amine of the formula

$$(C_{17}H_{33})N[(C_2H_4O)_{4.5}H]_2.$$

Dressing Leather. Brit. Pat. 822,231. Farbenfriken Bayer A.G. Appl. June 30, 1958.—Leather is dressed with dispersions of polymers containing carboxylic acid groups in the presence of a zinc compound. Suitable dispersions are those obtained by copolymerization of esters of acrylic or methacrylic acid, such as the methyl, ethyl, butyl or octyl esters, with vinyl chloride, vinylidene chloride, styrene, butadiene, vinyl alcohol derivatives, vinyl ethers, acrylonitrile, methacrylonitrile, acrylic acid amide, methacrylic acid amide, alkylvinyl ketones, arylvinyl ketones, vinyldicarboxylic acid esters such as diethylfumaric acid ester, or divinyl compounds such as divinyl benzene, or mono-functional and polyfunctional vinyl carboxylic acids such as acrylic acid, methacrylic acid, fumaric acid, maleic acid and hemi-esters of these dicarboxylic acids. Suitable zinc compounds are finely divided zinc oxide, zinc hydroxide or water-soluble zinc salts such as zinc acetate. The zinc compound should be used in quantity at least equivalent to the free carboxylic acid groups of the polymer dispersion used.

Coated Leather. Brit. Pat. 819,203. Lewis, J., and Rubber Improvement Ltd. Appl. July 26, 1956.

FIG. 1.

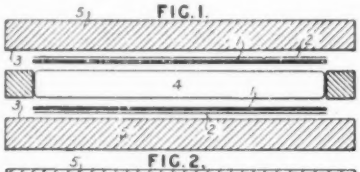
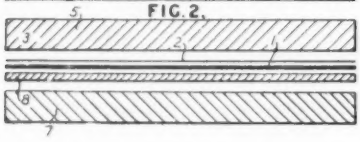


FIG. 2.



Leather is coated with film 2 of a vinyl polymer or copolymer which is applied as a paste consisting of the polymer or copolymer, plasticizers, stabilizers, and pigment with or without a solvent, and affixed by heat fusion under

pressure applied between the leather 1 and a plate matrix 3 by means of a resiliently compressible pressure member, e.g. a pad or sponge 8 supported by a platen 7, or a bag 4 which may be expanded by increasing the air or gas pressure within it, acting on the uncoated surface of the leather. The surface of the leather may be slightly buffed and may be given a preparatory coating with a latex of a vinyl polymer or copolymer containing a stabilizer and plasticizer. The coated leather may be heated and cooled prior to release of the pressure by means of press platen 5 and a glassy surface may be obtained by using a polished metal plate or matrix, e.g. of stainless steel. The vinyl polymer or copolymer may be one of a mixture of two or more of the substances: polyvinyl chloride, vinyl chloride-vinyl acetate copolymer, polyvinyl butyral and polyvinyl formal, which may be plasticized with dioctylphthalate, and stabilized with lead carbonate or stearate.

Proofing Against Fungi. Brit. Pat. 819,069. Pittsburgh Coke & Chemical Co. Appl. Dec. 5, 1955.—Cloth, leather or other fibrous materials or wood are proofed against fungi by treatment with a bis-(alkylsulphonyl)-ethylene of the formula $RSO_2CH{=}CHSO_2R$ where R is an alkyl group of 3 to 12 carbon atoms dissolved in a volatile solvent such as benzene, xylene, toluene, acetone, methyl ethyl ketone, cyclohexanone, carbon tetrachloride, perchloroethylene, trichloroethylene, ethyl, butyl and amyl acetate, ethanol, isopropanol, amyl alcohol and the monoalkyl ethers of the diethylene and ethylene glycols.

Process for Making Wool Resistant to Felting and Shrinking. U. S. Pat. 2,926,064. A. Rapp, Dusseldorf-Holthausen, Germany, assignor to Böhme Fettchemie G.m.b.H., Dusseldorf, Germany, a corporation of Germany. Appl. June 25, 1956.—1. The method of simultaneously felt- and shrink-proofing wool, which comprises immersing said wool in a single aqueous bath containing essentially between 0.1 and 1.0 gm./liter of a polymerized epoxide compound having an epoxide oxygen content from 2 to 10%, said epoxide compound selected from the group consisting of carboxylic acid epoxy-alkyl esters, epoxy-alkyl ethers and epoxidized unsaturated fatty oils, an inert solvent selected from the group consisting of tetrahydrofurfuryl alcohol, dioxane acetone and diacetone alcohol, an emulsifier selected from the group consisting of ethylene oxide addition products of fatty alcohols with 12 to 14 carbon atoms and ethylene oxide addition products of fatty acids with 12 to 18 carbon atoms, and a compound capable of releasing from 0.1 to 0.5 gm./liter of an element selected from the group consisting of chlorine and oxygen in aqueous solution, said compound selected from the group consisting of alkali metal hypochlorites, hypochlorous acid and persulfuric acid, allowing said wool to remain in said aqueous bath at substantially room temperature for a sufficient amount of time ranging from about 5 to about 10 minutes to become thoroughly impregnated with said aqueous bath, removing the wool from the aqueous bath and drying the impregnated wool at a temperature of about 70°C.

Process for Treating Protein with Pyrocarbonic Acid Esters. U. S. Pat. 2,920,068. H. Bernhard, Koln-Stammheim, O. Wahl, Opladen, and H. Genth, Krefeld-Bockum, Germany, assignors to Farbenfabriken Bayer Aktiengesellchaft, Leverkusen, Germany, a corporation of Germany. Appl. July 2, 1957.—1. A process for treating a protein selected from the group consisting of gelatin

and glue which comprises adding a pyrocarbonic acid ester gradually with stirring to a neutral aqueous solution of the protein at a temperature between 35 and 50°C., said pyrocarbonic acid ester having the general formula

wherein R_1 and R_2 each represent a member of the group consisting of an alkyl radical containing up to 5 carbon atoms and the cyclohexyl, benzyl, phenyl and furfuryl radical.

Collagen Article and the Manufacture Thereof. U. S. Pat. 2,920,000. H. R. Hochstadt, Franklin Park, and E. R. Lieberman, Somerville, N. J., assignors to Ethicon, Inc., a corporation of New Jersey. Appl. Oct. 22, 1958.—4. In a process for the extrusion of a homogeneous collagen dispersion to form a shaped article, the improvement which comprises swelling and dispersing collagen fibers derived from animal tendons in an aqueous solution of cyanoacetic acid.

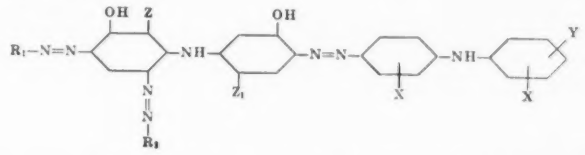
Method of Tanning. U. S. Pat. 2,923,594. E. R. Lieberman, Somerville, and P. V. Fagan, New Brunswick, N. J., assignors to Ethicon, Inc., a coporation of New Jersey. Appl. May 29, 1958.—1. In a method for preparing collagen sutures, the improvement which comprises treating catgut with an aqueous solution of an alkali metal bisulfite adduct of a compound selected from the group consisting of tolylene diisocyanate, hexamethylene diisocyanate and n-butylisocyanate at a temperature up to about 40°C.

Method of Soaking Animal Hides and Skins. U. S. Pat. 2,923,595. R. Heyden and J. Plapper, Dusseldorf, Germany, assignors to Böhme Fettchemie G.m.b.H., Dusseldorf, Germany, a corporation of Germany. Appl. Jan. 20, 1958. —1. The method of soaking raw animal hides and skins, which comprises immersing said hides and skins in an aqueous solution having dissolved therein from 0.05 to 1.00 gm./liter of compounds selected from the group consisting of mercapto-lower alkane-sulfonic acids and their water-soluble salts.

Method of Rendering Wool Unshrinkable and Non-Felting. U. S. Pat. 2,923,596. M. Levin, Jerusalem, Israel, assignor to The Ministry of Agriculture (The Institute for Fibres and Forest Products), Jerusalem, Israel, a ministry of the Government of Israel. Appl. Jan. 4, 1956.—1. A process for rendering wool shrinkproof and feltproof comprising immersing the wool in an aqueous acid-bromate solution containing bromate ions in an amount of 1.45 to 6.25% and a substance from the group consisting of alkali and alkaline-earth chlorides in an amount of from 20 to 120% (calculated as chloride ion), and a strong mineral acid in an amount at least equivalent to that of the bromate, all percentages being by weight of air-dry wool: and leaving the wool immersed in the solution for the time required for the disappearance from the solution of bromate ion in an amount of about 1.3 to 2.1% by weight of the air-dry wool, the irretrievable bromate ions being from 1.3 to 1.7 percent and the retrievable sorbed bromate ions being from 0 to 0.4 percent.

Method of Packaging and Treating Articles. U. S. Pat. 2,904,392. R. Pomerantz, Chicago, Ill., and E. F. Degering, Natick, and T. Hasselstrom, Framingham, Mass. Appl. April 19, 1956.—1. A method of preserving leather material with paranitrophenol as a fungus inhibitor, enclosing the treated material in a closed gas-impervious container formed from a thin transparent film composed of polymeric ethylene terephthalate, and exposing the resultant package to high intensity ionizing irradiation at a dosage level of at least about 0.5 megarep to effect sterilization.

Polyazo-Dyestuffs. U. S. Pat. 2,897,188. J. Weissert, Frankfurt am Main, and H. Gies, Hofheim (Taunus), Germany, assignors to Farbwerke Hoechst Aktiengesellschaft vormals Meister Lucius & Brusing, Frankfurt am Main, Germany. Appl. Nov. 21, 1957.—1. The polyayzo-dyestuffs corresponding to the following general formula:

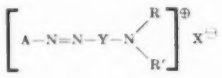


wherein one X stands for hydrogen and the other X stands for the sulfonic acid group, Y represents a member selected from the group consisting of hydrogen, nitro and acetylamino, Z stands for a member selected from the group consisting of hydrogen and the group N=N—R_3, Z_1 stands for a member selected from the group consisting of hydrogen and the group N=N—R_4, R_1 and R_2 represent members selected from the group consisting of radicals of the benzene and diphenylamine series, R_3 represents a radical of the diphenylamine series and R_4 represents a member selected from the group consisting of radicals of the benzene, naphthalene and diphenylamine series. Dyes all leathers, especially glove leather.

Collagen Article and the Manufacture Thereof. U. S. Pat. 2,919,998. H. B. Klevens, Pittsburgh, Pa., and J. Nichols, Princeton, N. J., assignors to Ethicon, Inc., a corporation of New Jersey. Appl. Nov. 7, 1957.—4. In a process for the extrusion of a collagen dispersion to form a shaped article, the improvement which comprises swelling and dispersing collagen fibers derived from animal tendons in an aqueous solution of a perfluoro acid having the formula $CF_3(CF_2)_nCOOH$, in which n is a whole integer smaller than 5.

Collagen Article and the Manufacture Thereof. U. S. Pat. 2,919,999. T. L. Reissmann, Bound Brook, and J. Nichols, Princeton, N. J., assignors to Ethicon, Inc., a corporation of New Jersey. Appl. Oct. 22, 1958.—1. In a process for the extrusion of a homogeneous collagen dispersion to form a shaped article, the improvement which comprises swelling and dispersing collagen fibers derived from animal tendons in an aqueous methanol solution of a perfluoro acid having the formula $CF_3(CF_2)_nCOOH$, in which n is a whole integer smaller than 7, said methanol amounting to about 50 percent of the total weight of the aqueous methanol solution.

Water-Soluble Azo Color Salts. U. S. Pat. 2,883,373. W. Bossard, Riehen, near Basel, and J. Voltz and F. Favre, Basel, Switzerland, assignors to J. R. Geigy A.-G., Basel, Switzerland, a Swiss firm. Appl. Jan. 23, 1956.—1. A dyestuff of the formula



the cation part of which is free from acid salt-forming groups and wherein A represents a radical selected from the group consisting of an N-lower alkyl cyclammonium ring system and an N-benzyl cyclammonium ring system, each comprising a single hetero nucleus of five ring members of which at least two are ring N-atoms, said radical being bound to the azo group by a C-atom that forms part of said hetero nucleus, Y represents a p-phenylene radical, R and R′ are members selected from the group consisting of hydrogen, lower aliphatic, benzyl and phenyl radicals and radicals wherein R and R′ are combined and together with the amino nitrogen atom form a heterocyclic ring, and X represents an anion. Suitable for dyeing leather.

Organosilicon-Chromium Coordination Complexes. U. S. Pat. 2,894,-967. J. W. Gilkey, Midland, Mich., assignor to Dow Corning Corporation, Midland, Mich., a corporation of Michigan. Appl. Aug. 2, 1956.—1. A composition comprising a coordination complex of the group consisting of (1) a complex formed by reacting $Cr(OH)Cl_2$ with an organosilicon compound containing units of the formula

$$R_nSi(R'COOH)O_{\frac{4-n}{2}}$$

where R is a monovalent hydrocarbon radical free of aliphatic unsaturation and having a maximum of 18 carbon atoms, R′ is a divalent alkylene radical of from 2 to 18 inclusive carbon atoms with the carboxyl group being at least 2 carbon atoms removed from the Si, and n is an integer of from 0–3 inclusive, and (2) the hydrolyzed and ammoniated derivatives thereof. Useful for waterproofing leather.

New Water-Soluble Complexes and a Process for Their Preparation. U. S. Pat. 2,904,572. X. Bilger, Soisy-sous-Montmorency, and J. Pechmeze, Paris, France, assignors to Compagnie Francaise des Matieres Colorantes, Paris, France, a French body corporate. Appl. Nov. 26, 1956.—1. A process for the preparation of water-soluble metallo-mixed complexes which comprises reacting in solution in an alcohol and at a temperature between 50°C. and 120°C. a basic chromic chloride with the copper salt of 8-hydroxy-quinoline. These Werner complexes are useful in leather treatment.

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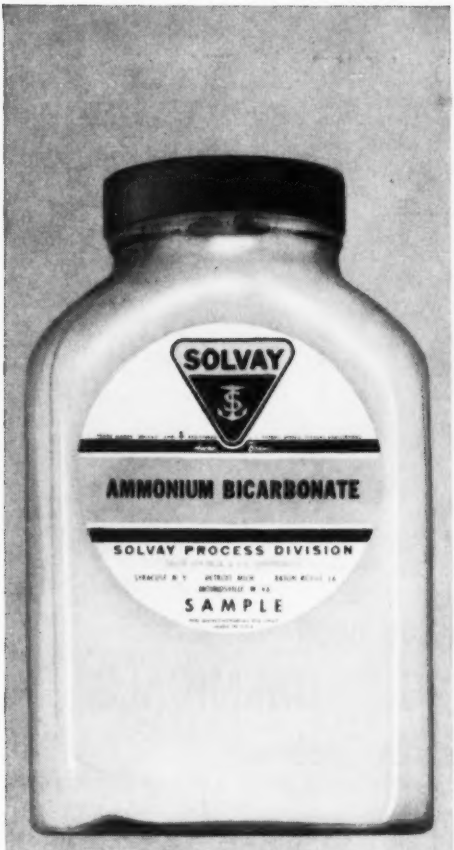

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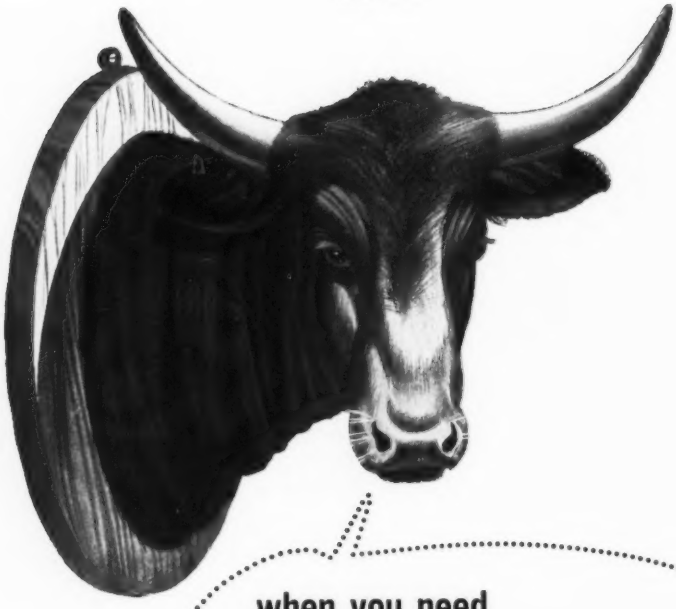

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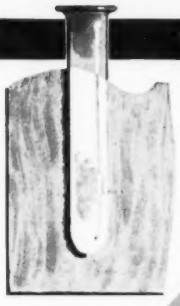

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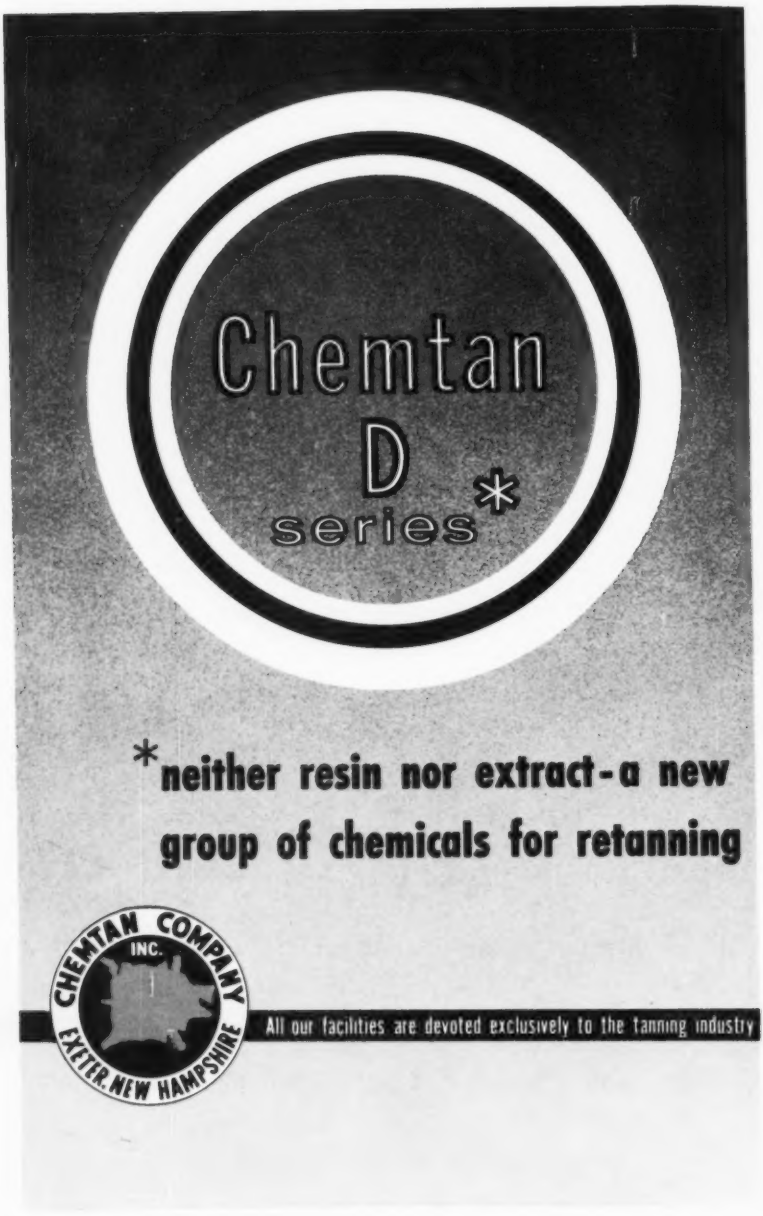
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